

State of California  
California Environmental Protection Agency  
AIR RESOURCES BOARD

**APPENDICES**

**FOR THE**

Report for the Application  
and Ambient Air Monitoring  
of Aldicarb

Engineering and Laboratory Branch

Monitoring and Laboratory Division

Project No. C97-007(b) (Application)  
C97-004(b) (Ambient)

Date: November 16, 1998

APPENDIX I  
SAMPLING PROTOCOL



**Cal/EPA**

California  
Environmental  
Protection  
Agency



**Air Resources Board**

P.O. Box 2815  
2020 L Street  
Sacramento, CA  
95812-2815

**MEMORANDUM**

*George  
C. Brown  
Kevin*



**Pete Wilson**  
Governor

**James M. Strock**  
Secretary for  
Environmental  
Protection

**TO:** John S. Sanders, Ph.D., Chief  
Environmental Monitoring and Pest  
Management Branch  
Department of Pesticide Regulation

**FROM:** George Lew, Chief *George Lew*  
Engineering and Laboratory Branch

**DATE:** March 21, 1997

**SUBJECT:** FINAL PROTOCOL FOR THE 1997 ALDICARB APPLICATION  
MONITORING IN FRESNO COUNTY

Enclosed is the final protocol, "Protocol for the Application Air Monitoring of Aldicarb in Fresno County During March, 1997."

If you or your staff have questions or need further information, please contact me at (916) 263-1630 or Mr. Kevin Mongar at (916) 263-2063.

Enclosure

**cc:** Ray Menebroker, Chief (w/Enclosure)  
Project Assessment Branch  
Stationary Source Division

**bcc:** Bill Loscutt, MLD  
Peter Venturini, SSD  
Don Ames, SSD  
Joan Denton, SSD  
Lynn Baker, SSD  
Doug Edwards, FC Ag. Comm. Office  
David Crow, SJVUAPCD

State of California  
California Environmental Protection Agency  
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Protocol for the Application Air Monitoring  
of Aldicarb  
In Fresno County During March, 1997

Engineering and Laboratory Branch

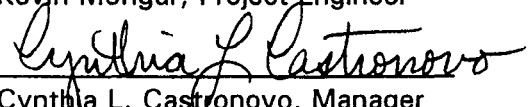
Monitoring and Laboratory Division

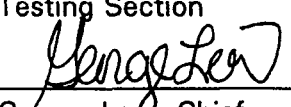
Project No. C97-007

Date: March 21, 1997

APPROVED:

\_\_\_\_\_  
Kevin Mongar, Project Engineer

  
\_\_\_\_\_  
Cynthia L. Castonovo, Manager  
Testing Section

  
\_\_\_\_\_  
George Lew, Chief  
Engineering and Laboratory Branch

This protocol has been reviewed by the staff of the California Air Resources Board and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Air Resources Board, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.



Protocol for the Application Air Monitoring  
of Aldicarb  
In Fresno County During March, 1997

I. Introduction

At the request of the California Department of Pesticide Regulation (DPR), (April 3, 1996 Memorandum from John Sanders to George Lew) the Air Resources Board (ARB) staff will determine airborne concentrations of the pesticide aldicarb (2-methyl-2-[methylthio]propionaldehyde O-methylcarbamoyloxime) over a 72 hour source impacted monitoring program at an agricultural application site. This monitoring will be done to fulfill the requirements of AB 1807/3219 (Food and Agricultural Code, Division 7, Chapter 3, Article 1.5) which requires the ARB "to document the level of airborne emissions .... of pesticides which may be determined to pose a present or potential hazard..." when requested by the DPR. The monitoring program will be conducted in Fresno County.

The draft method development results and "Standard Operating Procedures for the Analysis of Aldicarb in Ambient Air" (contracted to the California Department of Food and Agriculture, Center for Analytical Chemistry (CDFA)) are not included in this protocol but will be included in the draft report.

II. Chemical Properties of Aldicarb

Aldicarb (CAS: 116-063) is a colorless crystalline solid with a faint sulfurous odor. Aldicarb has a molecular formula of  $C_7H_{14}N_2O_2S$ , a molecular weight of 190.27, and a specific density of 1.195 at 25°C. It has a water solubility of 6.0 g/L at 25 °C, a Henry's Constant of  $1.45 \times 10^{-9}$  atm·m<sup>3</sup>/mol at 20-25 °C, and a vapor pressure of  $3.47 \times 10^{-5}$  mmHg at 25 °C. Aldicarb is miscible with most organic solvents.

In soil, aldicarb is rapidly converted to aldicarb sulfoxide in the presence of oxidizing agents and microorganisms. Further oxidation to the sulfone by microorganisms occurs at a slower rate. Mineralization was more rapid in aerobic surface soils than either aerobic or anaerobic subsurface soils; aldicarb degrades rapidly in aerobic silty clay loam soils releasing carbon dioxide. Reported soil half-life ( $t_{1/2}$ ) ranges from 7 to 70 days. In water, the reported hydrolysis half-lives range from 175 to 245 days. In plants, with the exception of cotton, aldicarb is rapidly metabolized to aldicarb sulfoxide, sulfone, and water-soluble noncarbamate compounds. Aldicarb sulfoxide is highly soluble, acts systemically on the plant, and is 10-20 times more active as a cholinesterase inhibitor than the parent compound.

The acute oral LD<sub>50</sub> of aldicarb for rats ranges from 650 to 930 µg/kg. The LC<sub>50</sub> (96 hour) of aldicarb for rainbow trout is 0.88 mg/L, and for bluegill sunfish is 1.5 mg/L. The LC<sub>50</sub> (72 hour) for bluegill sunfish is 100 µg/L. Aldicarb has entered the risk assessment process at DPR under the Birth Defect Prevention Act of 1984 (SB950) primarily because of its cholinesterase inhibition.

### III. Sampling

Samples will be collected by passing a measured volume of ambient air through XAD-2 resin. The exposed XAD-2 resin tubes (SKC #226-30-06) are stored in an ice chest (dry ice) or freezer until desorbed with 3 ml of ethyl acetate. The flow rate will be accurately measured and the sampling system operated continuously with the exact operating interval noted. The resin tubes will be protected from direct sunlight and supported about 1.5 meters above the ground during the sampling period. At the end of each sampling period, the tubes will be capped and placed in culture tubes with an identification label affixed. Any aldicarb present in the sampled ambient air will be captured by the XAD-2 adsorbent. Subsequent to sampling, the sample cartridges will be transported on dry ice, as soon as reasonably possible, to the California Department of Food and Agriculture laboratory for analysis. The samples will be stored in the freezer (-20 C) or analyzed immediately.

A sketch of the sampling apparatus is shown in Attachment A. Calibrated rotameters will be used to set and measure sample flow rates. Samplers will be leak checked prior to and after each sampling period with the sampling cartridges installed. Any change in the flow rates will be recorded in the field log book. The field log book will also be used to record start and stop times, sample identifications and any other significant data.

#### Application Monitoring

The use pattern for aldicarb suggests that application-site monitoring may be conducted during the week of March 24, 1997 in Fresno County, and that the application be associated with cotton. Due to the extensive use of aldicarb during this period, care should be taken so that other applications to nearby fields during the sampling period do not affect sample collection. A three day monitoring period should be established with sampling times as follows; (where the first sample is started at the start of application) application + 1 hour, followed by one 2-hour sample, one 4-hour sample, two 8-hour samples and two 24-hour samples. A minimum of four samplers should be positioned, one on each side of the field. A fifth sampler should be collocated at one position. Ideally samplers should be placed a minimum of 20 meters from the field with a sampling intake approximately 1.5 meters above the ground. Prior to application, background samples will be taken to establish if any aldicarb is detectable. Since aldicarb is extensively used in the area, background samples should collect enough volume (either 12 hours at 15 liters/min., or a shorter period with a higher volume pump) to permit a reasonable minimum detection level. A meteorological station will be set up to determine wind speed and direction. This station will continue to operate continuously throughout the sampling period collecting data at a minimum of 15 minute intervals using a data logger. ARB staff will note the degree of cloud cover at the start of application and whenever sample cartridges are changed. Air samples will be collected with XAD-2 resin using battery powered pumps capable of flows of approximately 4 liters per minute.

The sampling location for the application monitoring has not yet been determined. The site will be chosen with close coordination between ARB staff, the Fresno County Agricultural Commissioner's office and local pesticide applicators.

#### IV. Analysis

The method development results and "Standard Operating Procedures for the Analysis of Aldicarb in Ambient Air" are not included in this protocol but will be included in the draft report.

#### V. Quality Assurance

Field Quality Control for the ambient monitoring will include: 1) Five field spikes (sample environmental and experimental conditions as those occurring at the time of ambient sampling) will be prepared and spiked at five different levels. The field spikes will be obtained by sampling ambient air at the background monitoring site for 24 hour periods at 4 L/minute. 2) Five trip spikes will be prepared and spiked at five different levels. 3) Replicate samples will be taken for five dates at each sampling location. 4) Trip blanks will be obtained at each of the five sampling locations.

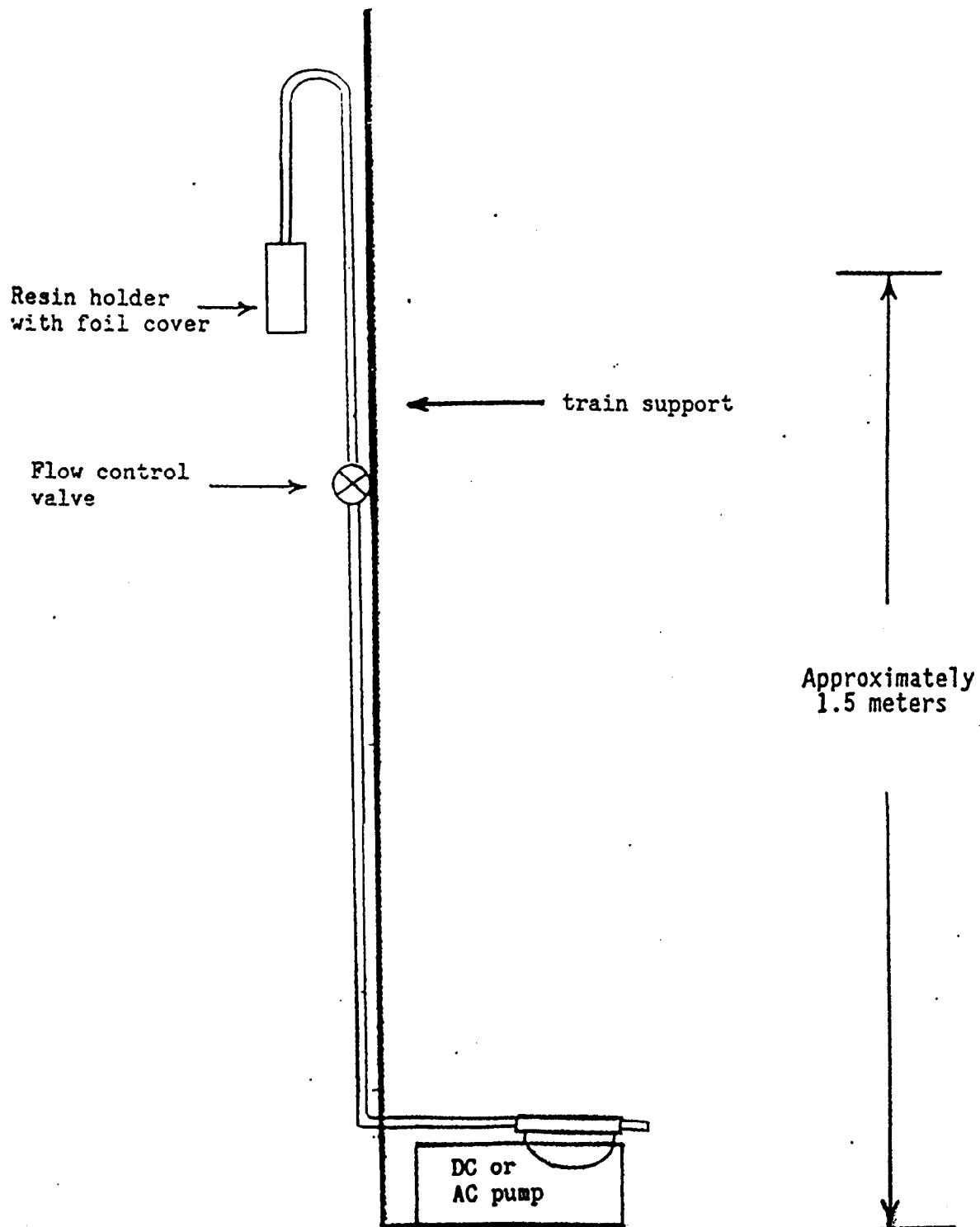
A chain of custody sheet will accompany all samples. Rotameters will be calibrated prior to and after sampling in the field.

#### VI. Personnel

ARB personnel will consist of Kevin Mongar (Project Engineer) and an Instrument Technician.

**Attachment A**

FIGURE 1  
FIELD SAMPLING APPARATUS



State of California  
California Environmental Protection Agency  
AIR RESOURCES BOARD

Protocol for the Ambient Air Monitoring  
of Aldicarb  
In Fresno County During April, 1997

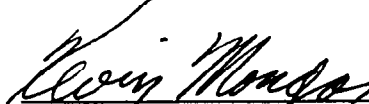
Engineering and Laboratory Branch

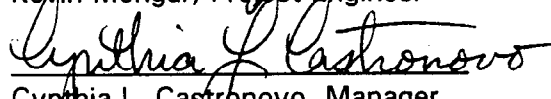
Monitoring and Laboratory Division

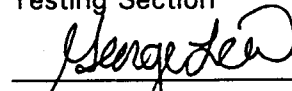
Project No. C97-004

Date: March 18, 1997

APPROVED:

  
Kevin Mongar, Project Engineer

  
Cynthia L. Castronovo, Manager  
Testing Section

  
George Lew, Chief  
Engineering and Laboratory Branch

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Protocol for the Ambient Air Monitoring  
of Aldicarb  
In Fresno County During April, 1997

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A sketch of the sampling apparatus is shown in Attachment A. Calibrated rotameters will be used to set and measure sample flow rates. Samplers will be leak checked prior to and after each sampling period with the sampling cartridges installed. Any change in the flow rates will be recorded in the field log book. The field log book will also be used to record start and stop times, sample identifications and any other significant data.

#### Ambient Monitoring

The historical trends in aldicarb use suggest that monitoring should occur over a 30- to 45-day sampling period in Kern, Kings or Fresno Counties from late March through early May, with the bulk of the sampling conducted in April. Three to five sampling sites should be selected in relatively high-population areas or in areas frequented by people. Sampling sites should be located near cotton growing areas. Ambient samples should not be collected from samplers immediately adjacent to fields or orchards where aldicarb is being applied. At each site, twenty to thirty discrete 24-hour samples should be taken during the sampling period. Background samples should be collected in an area distant to aldicarb applications.

Replicate (collocated) samples are needed for five dates at each sampling location. Two collocated samplers (in addition to the primary sampler) should be run on those days. The dates chosen for replicate samples should be distributed over the entire sampling period. They may, but need not be, the same dates at every site. Field spike samples should be collected at the same environmental conditions (e.g., temperature, humidity, exposure to sunlight) and experimental conditions (e.g., air flow rates) as those occurring at the time of ambient sampling.

Four sampling sites plus an urban background site were selected by ARB personnel from the areas of Fresno County where cotton farming is predominant. Sites were selected for their proximity to the cotton fields with considerations for both accessibility and security of the sampling equipment. The five sites were at the following locations: Addresses for the sites are listed in Table 1.



TABLE 1. Ambient Sampling Sites		
BOR	Burrell Elementary School 16704 South Jameson Burrell, CA 93607	(209) 866-5634 Mildred Wylie Principal
ARB	Air Resources Board, Ambient Air Monitoring Station 3425 N First, Suite 205B Fresno, CA 228-1825	(209) 228-1825
FP	Westside Elementary School 19191 Excelsior Ave. Five Points, CA 93624	(209) 884-2492 Baldomero Hernandez Principal
SJ	San Joaquin Elementary School 8535 South 9th San Joaquin, CA 93660	(209) 693-4321 Jackie Newman Principal
HEL	Helm Elementary School 13883 S. Lassen Avenue Helm, CA 93627	(209) 866-5683 Sylvia Grider

The samples will be collected by ARB personnel over a six week period from March 24 - May 2, 1997. 24-hour samples will be taken Monday through Friday (4 samples/week) at a flow rate of 4.0 L/minute.

#### IV. Analysis

The method development results and "Standard Operating Procedures for the Analysis of Aldicarb in Ambient Air" are not included in this protocol but will be included in the draft report.

#### V. Quality Assurance

Field Quality Control for the ambient monitoring will include:

- 1) Five field spikes (same environmental and experimental conditions as those occurring at the time of ambient sampling), spiked at five different levels. The field spikes will be obtained by sampling ambient air at the background monitoring site for 24 hour periods at 4.0 L/minute.
- 2) Five trip spikes will be prepared and spiked at five different levels.
- 3) Replicate samples will be taken for six dates at each sampling location.

- 4) Trip blanks will be obtained at each of the five sampling locations.

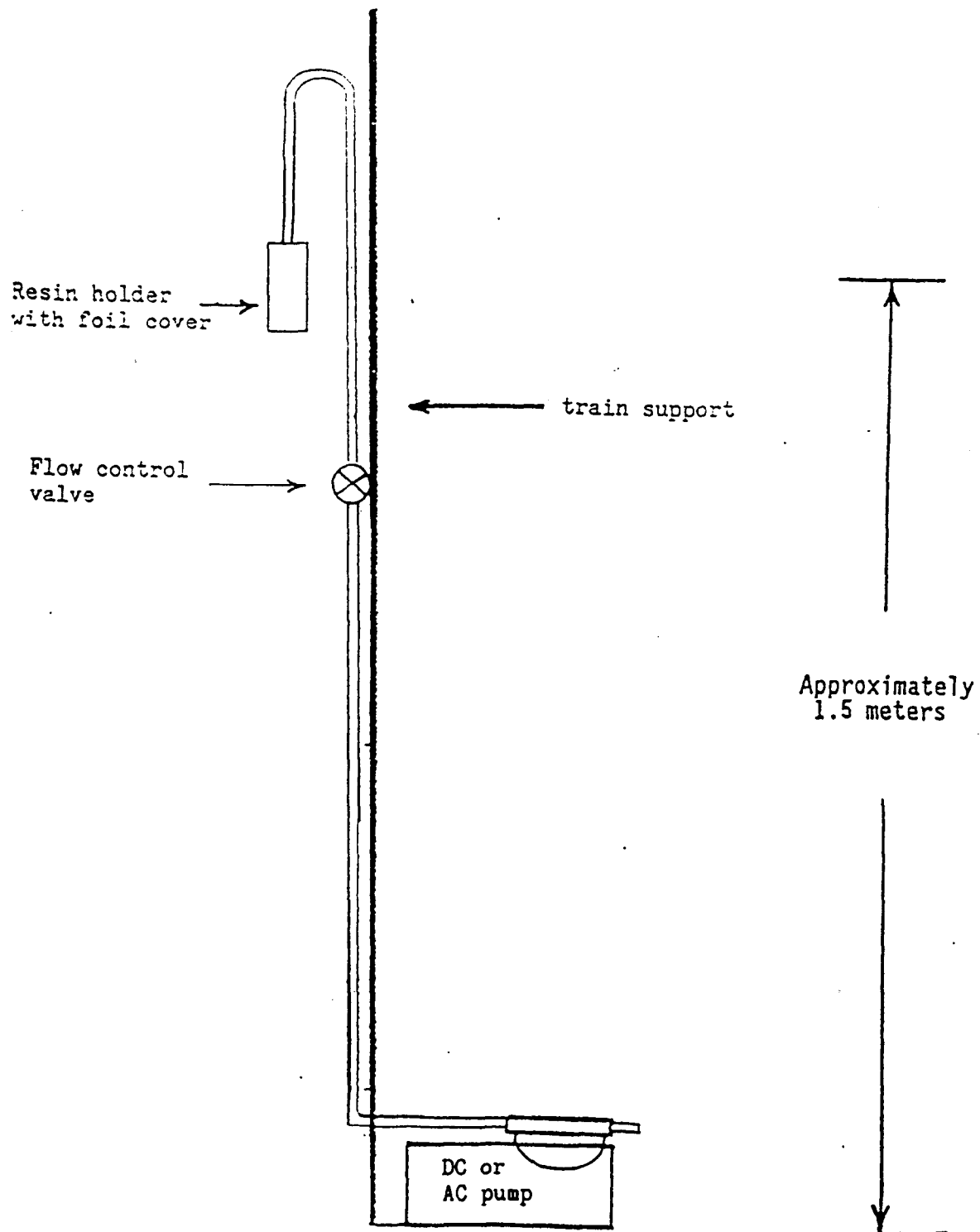
A chain of custody sheet will accompany all samples. Rotameters will be calibrated prior to and after sampling in the field.

VI. Personnel

ARB personnel will consist of Kevin Mongar (Project Engineer) and an Instrument Technician.

**Attachment A**

FIGURE 1  
FIELD SAMPLING APPARATUS



APPENDIX II

LABORATORY REPORT

Worker Health and Safety  
Laboratory



Center for Analytical Chemistry  
3292 Meadowview Road  
Sacramento, California  
916-262-2079

Air Sample Analysis Report  
for  
Aldicarb Application and Aldicarb Ambient



Submitted by:  
Sheila Margetich  
Supervisor  
Worker Health and Safety Laboratory  
9-29-97

## Table of Contents

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I. Summary of ARB/CAC Contract	1
Table #1 - ARB Air Sample Log	
II. Analytical Results Record	2-9
Table #2	
III. Summary of WHS Analytical Report	10-16
Table #3 - Application QA Spikes % Recovery	13
Table #4 - Ambient QA Spikes % Recovery	14
Table #5 - WHS On-Going QC Spikes % Recovery	15
Table #6 - WHS On-Going QC Spikes % Recovery	16
Table #7 - WHS On-Going QC Blank Results	16
IV. Attachments	
A. ARB Original Chain of Custody Forms	
B. Method SOP	
C. Chromatogram of Standard at LOD Concentration	
D. Chromatograms of Standard Curve Concentrations	
E. WHS-AD-11 SOP Titled "Data Generation and Reporting"	
F. Resin Lab, Field and Trip Spike Chromatograms	
G. Chromatogram of On-Going Resin QC Spike	
H. Chromatogram of On-Going Resin QC Blank Results	
I. Chromatogram of Resin Air Sample	
J. Method Validation Report	



## I. Summary of ARB/CAC Contract

The Worker Health and Safety Laboratory (WHS) of the Center for Analytical Chemistry (CAC) was contracted by the Air Resources Board to perform the analysis of air samples. In partial agreement of that contract, we analyzed 6 sets of Aldicarb ambient samples, 1 set of Aldicarb application samples and accompanying QA samples. Method development and method validation was required for this analyte and its metabolites.

The following table summarizes the samples submitted by ARB for analyses and their analytical completion dates. Please see Attachment A1 > A 20 for the original chain of custody forms that accompanied these samples.

**TABLE 1. ARB AIR SAMPLE LOG WITH ANALYTICAL COMPLETION DATES**

Date Received	ARB Logbook Numbers (Inclusive)	Total # of air samples	Analysis Completion Date
3-28-97	Aldicarb Ambient 1-26	26	4-28-97
3-28-97	Aldicarb Application 1-38	30 + 8 QA spikes	4-17-97
4-4-97	Aldicarb Ambient 27-52	26	4-30-97
4-11-97	Aldicarb Ambient 53-78	26	5-6-97
6-13-97	Aldicarb Ambient 79-99	21	6-23-97
6-20-97	Aldicarb Ambient 100-125	26	6-30-97
6-27-97	Aldicarb Ambient 126-161	26 + 10 QA spikes	7-8-97

TABLE 2.

## ANALYTICAL RESULT RECORD

ARB Aldicarb Ambient Logbook Number	ARB Field Sample Number	Aldicarb Results * ug/sample	WHS Lab Number
1	SJ-1	ND	WHSC-1
2	HEL-1	ND	WHSC-2
3	FP-1	ND	WHSC-3
4	BOR-1	ND	WHSC-4
5	ARB-1	ND	WHSC-5
6	SJ-2	ND	WHSC-6
7	HEL-2	ND	WHSC-7
8	FP-2	ND	WHSC-8
9	BOR-2	ND	WHSC-9
10	ARB-2	ND	WHSC-10
11	SJ-3	ND	WHSC-11
12	SJ-3D	ND	WHSC-12
13	HEL-3	ND	WHSC-13
14	HEL-3D	ND	WHSC-14
15	FP-3	ND	WHSC-15
16	FP-3D	ND	WHSC-16
17	BOR-3	ND	WHSC-17
18	BOR-3D	ND	WHSC-18
19	ARB-3	ND	WHSC-19
20	ARB-3D	ND	WHSC-20
21	B-3	ND	WHSC-21
22	SJ-4	ND	WHSC-22
23	HEL-4	ND	WHSC-23
24	FP-4	ND	WHSC-24
25	BOR-4	ND	WHSC-25
26	ARB-4	ND	WHSC-26

\* Aldicarb Limit of Detection: 0.05 ug/sample

TABLE 2.

ANALYTICAL RESULT RECORD

ARB Aldicarb Ambient Logbook Number	ARB Field Sample Number	Aldicarb Results * ug/sample	WHS Lab Number
27	SJ-5	ND	WHSC-57
28	HEL-5	ND	WHSC-58
29	FP-5	ND	WHSC-59
30	BOR-5	ND	WHSC-60
31	ARB-5	ND	WHSC-61
32	SJ-6	ND	WHSC-62
33	HEL-6	ND	WHSC-63
34	FP-6	ND	WHSC-64
35	BOR-6	ND	WHSC-65
36	ARB-6	ND	WHSC-66
37	SJ-7	ND	WHSC-67
38	SJ-7D	ND	WHSC-68
39	HEL-7	ND	WHSC-69
40	HEL-7D	ND	WHSC-70
41	FP-7	ND	WHSC-71
42	FP-7D	ND	WHSC-72
43	BOR-7	ND	WHSC-73
44	BOR-7D	ND	WHSC-74
45	ARB-7	ND	WHSC-75
46	ARB-7D	ND	WHSC-76
47	Blank	ND	WHSC-77
48	SJ-8	ND	WHSC-78
49	HEL-8	ND	WHSC-79
50	FP-8	ND	WHSC-80
51	BOR-8	ND	WHSC-81
52	ARB	ND	WHSC-82

\* Aldicarb Limit of Detection: 0.05 ug/sample

(3)

TABLE 2.

ANALYTICAL RESULT RECORD

ARB Aldicarb Ambient Logbook Number	ARB Field Sample Number	Aldicarb Results * ug/sample	WHS Lab Number
53	SJ-9	ND	WHSC-126
54	HEL-9	ND	WHSC-127
55	FP-9	ND	WHSC-128
56	BOR-9	ND	WHSC-129
57	ARB-9	ND	WHSC-130
58	SJ-10	ND	WHSC-131
59	HEL-10	ND	WHSC-132
60	FP-10	ND	WHSC-133
61	BOR-10	ND	WHSC-134
62	ARB-10	ND	WHSC-135
63	SJ-11	ND	WHSC-136
64	SJ-11D	ND	WHSC-137
65	HEL-11	ND	WHSC-138
66	HEL-11D	ND	WHSC-139
67	FP-11	ND	WHSC-140
68	FP-11D	ND	WHSC-141
69	BOR-11	ND	WHSC-142
70	BOR-11D	ND	WHSC-143
71	ARB-11	ND	WHSC-144
72	ARB-11D	ND	WHSC-145
73	SJ-12	ND	WHSC-146
74	HEL-12	ND	WHSC-147
75	FP-12	ND	WHSC-148
76	BOR-12	ND	WHSC-149
77	ARB-12	ND	WHSC-50
78	Blank	ND	WHSC-151

\* Aldicarb Limit of Detection: 0.05 ug/sample (4)

TABLE 2.

## ANALYTICAL RESULT RECORD

ARB Aldicarb Application Logbook Number	ARB Field Sample Number	Aldicarb Results * ug/sample	WHS Lab Number
1	ALD-W1	ND	WHSC-27
2	ALD-N1	ND	WHSC-28
3	ALD-E1	ND	WHSC-29
4	ALD-E1D	ND	WHSC-30
5	ALD-S1	0.07	WHSC-31
6	ALD-B1	ND	WHSC-32
7	ALD-S2	ND	WHSC-33
8	ALD-E2	ND	WHSC-34
9	ALD-E2D	ND	WHSC-35
10	ALD-N2	ND	WHSC-36
11	ALD-W2	ND	WHSC-37
12	ALD-B2	ND	WHSC-38
13	ALD-S3	ND	WHSC-39
14	ALD-E3	ND	WHSC-40
15	ALD-E3D	ND	WHSC-41
16	ALD-N3	ND	WHSC-42
17	ALD-W3	ND	WHSC-43
18	ALD-B3	ND	WHSC-44
20	ALD-S4	0.06	WHSC-45
21	ALD-E4	ND	WHSC-46
22	ALD-E4D	ND	WHSC-47
24	ALD-N4	ND	WHSC-48
26	ALD-W4	ND	WHSC-49
28	ALD-B4	ND	WHSC-50
33	ALD-S5	ND	WHSC-51
34	ALD-E5	ND	WHSC-52

\* Aldicarb Limit of Detection: 0.05 ug/sample

(5)

ANALYTICAL RESULT RECORD

[illegible]

(6)

TABLE 2.

## ANALYTICAL RESULT RECORD

ARB Aldicarb Ambient Logbook Number	ARB Field Sample Number	Aldicarb Results * ug/sample	WHS Lab Number
79	WAS-1	ND	WHSC-187
80	MCF-1	ND	WHSC-188
81	MAP-1	ND	WHSC-189
82	SEV-1	ND	WHSC-190
83	BAK-1	ND	WHSC-191
84	WAS-2	ND	WHSC-192
85	WAS-2D	ND	WHSC-193
86	MCF-2	ND	WHSC-194
87	MCF-2D	ND	WHSC-195
88	MAP-2	ND	WHSC-196
89	MAP-2D	ND	WHSC-197
90	SEV-2	ND	WHSC-198
91	SEV-2D	ND	WHSC-199
92	BAK-2	ND	WHSC-200
93	BAK-2D	ND	WHSC-201
94	BLANK	ND	WHSC-202
95	WAS-3	ND	WHSC-203
96	MCF-3	ND	WHSC-204
97	MAP-3	ND	WHSC-205
98	SEV-3	ND	WHSC-206
99	BAK-3	ND	WHSC-207
100	MCF-4	ND	WHSC-208
101	WAS-4	ND	WHSC-209
102	MAP-4	ND	WHSC-210
103	SEV-4	ND	WHSC-211
104	BAK-4	ND	WHSC-212

\* Aldicarb Limit of Detection: 0.05 ug/sample

TABLE 2.

## ANALYTICAL RESULT RECORD

ARB Aldicarb Ambient Logbook Number	ARB Field Sample Number	Aldicarb Results * ug/sample	WHS Lab Number
105	MCF-5	ND	WHSC-213
106	WAS-5	ND	WHSC-214
107	MAP-5	ND	WHSC-215
108	SEV-5	ND	WHSC-216
109	BAK-5	ND	WHSC-217
110	MCF-6	ND	WHSC-218
111	MCF-6D	ND	WHSC-219
112	WAS-6	ND	WHSC-220
113	WAS-6D	ND	WHSC-221
114	MAP-6	ND	WHSC-222
115	MAP-6D	ND	WHSC-223
116	SEV-6	ND	WHSC-224
117	SEV-6D	ND	WHSC-225
118	BAK-6	ND	WHSC-226
119	BAK-6D	ND	WHSC-227
120	MCF-7	ND	WHSC-228
121	WAS-7	ND	WHSC-229
122	MAP-7	ND	WHSC-230
123	SEV-7	ND	WHSC-231
124	BAK-7	ND	WHSC-232
125	BLANK-2	ND	WHSC-233
126	MCF-8	ND	WHSC-234
127	WAS-8	ND	WHSC-235
128	MAP-8	ND	WHSC-236
129	SEV-8	ND	WHSC-237
130	BAK-8	ND	WHSC-238

\* Aldicarb Limit of Detection: 0.05 ug/sample

(8)



TABLE 2.

ANALYTICAL RESULT RECORD

ARB Aldicarb Ambient Logbook Number	ARB Field Sample Number	Aldicarb Results * ug/sample	WHS Lab Number
138	MCF-9	ND	WHSC-239
139	WAS-9	ND	WHSC-240
140	MAP-9	ND	WHSC-241
141	SEV-9	ND	WHSC-242
142	BAK-9	ND	WHSC-243
145	MCF-10	ND	WHSC-244
146	MCF-10D	ND	WHSC-245
147	WAS-10	ND	WHSC-246
148	WAS-10D	ND	WHSC-247
149	MAP-10	ND	WHSC-248
150	MAP-10D	ND	WHSC-249
151	SEV-10	ND	WHSC-250
152	SEV-10D	ND	WHSC-251
153	BAK-10	ND	WHSC-252
154	BAK-10D	ND	WHSC-253
155	BLANK	ND	WHSC-254
157	MCF-11	ND	WHSC-255
158	WAS-11	ND	WHSC-256
159	MAP-11	ND	WHSC-257
160	SEV-11	ND	WHSC-258
161	BAK-11	ND	WHSC-259

\* Aldicarb Limit of Detection: 0.05 ug/sample

### III. Summary of WHS Analytical Report

#### 1. SCOPE:

This report covers the WHS analysis of samples labeled Aldicarb Application Log #1-38, Aldicarb Ambient Log #1-161 and related QA samples.

#### 2. SUMMARY OF METHOD:

The analytical method titled "Standard Operating Procedure for the Analysis of Aldicarb and Metabolites on XAD-2 Resin Air Sampling Tubes" as developed and validated by Worker Health and Safety was followed with three exceptions: 1) halfway through the project we had trouble with the Spectra Physics detector and it was replaced with a Hewlett-Packard detector and connected to the HP ChemStation, 2) per ARB, all "None Detected" results were based on the method LOD rather than the method LOQ and 3) to lower the sensitivity, the method was revised to have a final volume of 0.667 mL for the samples. The method LOD was verified by analyzing 5 seasoned spikes at the 0.050 ug/spl level. Please see Attachment B for the Method SOP.

#### WHS Instrumentation

Hewlett Packard 1050 HPLC Liquid Chromatograph

Column: Pickering Laboratories Carbamate Analysis Column, 5  $\mu$ m, C<sub>18</sub>, 4.6 x 250 mm

Post Column System: Pickering PCX 5100

Pump Gradient: ACN/H<sub>2</sub>O - 10%, hold for 2 min., to 90% in 16 min, hold for 2 min, to 10% at 1.0 L/min.

Fluorescence Detector: Spectra Physics Thermo Separation  
excitation at 330 nm  
emission at 464 nm

Retention Times: Aldicarb Sulfoxide	6.50 min.
Aldicarb Sulfone	7.68 min.
Aldicarb	11.87 min.

### 3. ANALYTICAL CALCULATIONS:

A. Calculation of LOD: The Limit of Detection (LOD) for Aldicarb, Aldicarb Sulfoxide and Aldicarb Sulfone was based on at least a 3:1 signal to noise ratio for each of the three peaks. The average peak height for each of these three peaks was 3 mm. At this height, the LOD for Aldicarb was 0.010 µg/spl. The LOD for Aldicarb SO was 0.015 µg/spl. The LOD for Aldicarb SO<sub>2</sub> was 0.025 µg/spl. These LOD calculations were based on a 0.667 mL final volume for each of the ARB samples.

During method development, "seasoned" Aldicarb spikes were prepared and analyzed. These were resin tubes that were spiked with a liquid injection of Aldicarb only and then had air drawn through them for 24 hours at 4L/min. When analyzed, these "seasoned" tubes resulted in both metabolites being present in addition to the Aldicarb parent. Consequently, the LOD for an actual ARB sample was reported as the combined LODs for Aldicarb and its metabolites, i.e. LOD = 0.050 µg/sample.

Calculation of Aldicarb (only) LOD is as follows:

$$\frac{\text{LOD peak height}}{\text{standard peak height}} \times \mu\text{g standard injected} \times \text{uL stand. inj.} \times \frac{\text{sample final volume}}{\mu\text{L sample injected}} = \mu\text{g/sample}$$

#### EXAMPLE:

$$\frac{3 \text{ mm}}{5 \text{ mm}} \times \frac{0.025 \mu\text{g Ald. standard injected}}{\text{mL}} \times 40 \text{ uL stand. inj} \times \frac{0.667 \text{ mL final volume}}{40 \mu\text{L injected}} = 0.010 \mu\text{g/spl}$$

B. Analytical verification of "seasoned" LOD: Five resin tubes (SKC lot # 499) were each spiked by liquid injection with 0.050 µg of Aldicarb (only). The tubes had air drawn through them at 4 L/minute for 24 hours after which they were extracted. Please see Attachment C for the chromatogram of a "seasoned" Aldicarb spike at the 0.050 µg/spl LOD level.

### 3. ANALYTICAL CALCULATIONS: (cont.)

C. Sample result calculations: For reporting of sample results, the total micrograms of Aldicarb and the total micrograms of the two metabolites (corrected for molecular weights) were combined and reported as Aldicarb. Results are reported as total micrograms of Aldicarb per sample. Sample calculation is as follows:

Aldicarb:

$$\frac{3.5\text{mm PH spl}}{3\text{mm PH Ald}} \times \frac{0.025 \mu\text{g Ald.}}{\text{mL}} \times 40 \text{ uL Ald. inj.} \times \frac{1.0 \text{ mL}}{1000 \text{ uL}} \times \frac{0.667 \text{ mL}}{40 \text{ uL spl inj.}} \times \frac{1.0 \text{ mL}}{1000 \text{ uL}} = 0.019 \mu\text{g/sample}$$

Aldicarb SO:

$$\frac{3.5\text{mm PH spl}}{3\text{mm PH Ald SO}} \times \frac{0.025 \mu\text{g SO}}{\text{mL}} \times 40 \text{ uL Ald. inj.} \times \frac{1.0 \text{ mL}}{1000 \text{ uL}} \times \frac{0.667 \text{ mL}}{40 \text{ uL spl inj.}} \times \frac{1.0 \text{ mL}}{1000 \text{ uL}} = 0.019 \mu\text{g/spl SO}$$

$$0.019 \mu\text{g/spl SO} \times \frac{190.25 \text{ M.W. Ald.}}{206.25 \text{ M.W. Ald. SO}} = 0.017 \mu\text{g/sample Aldicarb}$$

Aldicarb SO<sub>2</sub>:

$$\frac{3.5\text{mm PH spl}}{3\text{mm PH Ald SO}_2} \times \frac{0.025 \mu\text{g SO}_2}{\text{mL}} \times 40 \text{ uL Ald. inj.} \times \frac{1.0 \text{ mL}}{1000 \text{ uL}} \times \frac{0.667 \text{ mL}}{40 \text{ uL spl inj.}} \times \frac{1.0 \text{ mL}}{1000 \text{ uL}} = 0.019 \mu\text{g/spl SO}_2$$

$$0.019 \mu\text{g/spl SO}_2 \times \frac{190.25 \text{ M.W. Ald.}}{222.25 \text{ M.W. Ald. SO}_2} = 0.016 \mu\text{g/sample Aldicarb}$$

TOTAL ALDICARB = 0.052  $\mu\text{g/sample}$

Where "PH" = Peak Height

### 4. QUALITY ASSURANCE:

A. Instrument Linearity and Reproducibility: Instrument linearity and reproducibility were evaluated by running 10 replicate 30ul injections of three levels of standards containing Aldicarb, SO and SO<sub>2</sub> over a 13 hour period. The spreadsheets of the ten standard curves are found as attachments to the Method SOP (Attachment B).

B. Standard Curve Linearity and r-value: A three point calibration curve, including 0.05  $\mu\text{g/mL}$ , 0.10  $\mu\text{g/mL}$  and 1.0  $\mu\text{g/mL}$  of Aldicarb, SO and SO<sub>2</sub>, was made to determine the standard curve linearity. Computed r-values for the 10 standard curves were also performed and the plots can be found as attachments in the Method SOP (Attachment B). Before the beginning of sample analysis, a four point calibration curve was established. A standard at the 0.025  $\mu\text{g/mL}$  level was added to cover the lower reportable LOD that was requested by the Study Director. Please see Attachment D1 > D4 for chromatograms of the standard curve levels.

#### 4. QUALITY ASSURANCE: (cont.)

C. Analytical result acceptance criteria: Analytical acceptance criteria based on the linearity and reproducibility of standard curves are detailed in Attachment E, our SOP numbered WHS-AD-11 and titled "Data Generation and Reporting".

##### D. Quality Assurance Spikes:

1. Application QA Spikes: The Center for Analytical Chemistry (CAC) Quality Assurance (QA) personnel prepared the Quality Assurance spikes for this portion of the study. The resin beds of sixteen resin tubes were spiked with 70 uL of 10 ng/uL Aldicarb spike solution. This made a total of 700 ng/spike. The standard was secured from CAC Standards Repository. The Standard Solution # was 72-2922e. After spiking, the tubes were allowed to stand at ambient temperature for about one hour after which the broken ends of the primary sections were capped.

Four tubes were selected at random, extracted and analyzed for spiking level verification. Four additional tubes were retained in the lab in Freezer # 27873 as Lab Spikes. The remaining eight tubes were used by ARB staff as Trip Spikes and Field Spikes. When the tubes were returned to the lab, all 12 tubes were extracted and analyzed concurrently. The following table lists the % recoveries.

TABLE 3. APPLICATION QA SPIKES - % RECOVERY

CAC Spike ID	ARB Spike ID	Amount Spiked ng	% Recovery Aldicarb	% Recovery Sulfoxide	% Recovery Sulfone	% Recovery Total Aldicarb
QA-LS-1	---	700	84.75	---	---	84.75
QA-LS-2	---	700	86.25	---	---	86.25
QA-LS-3	---	700	84.83	---	---	84.83
QA-LS-4	---	700	86.08	---	---	86.08
QA-TS-1	TS-1	700	76.00	1.97	---	77.82*
QA-TS-2	TS-2	700	76.34	14.12	4.63	93.32*
QA-TS-3	TS-3	700	69.03	3.26	---	72.04*
QA-TS-4	TS-4	700	73.12	3.09	---	75.97*
QA-FS-1	FS1S-4	700	33.24	58.68	9.97	95.90*
QA-FS-2	FS2N-4	700	41.16	47.71	12.01	95.45*
QA-FS-3	FS3W-4	700	31.62	50.30	13.54	89.61*
QA-FS-4	FS4B-4	700	25.17	47.23	11.81	78.84*

\* Total Aldicarb recoveries include Aldicarb metabolite amounts that were corrected for molecular weights.

#### 4. QUALITY ASSURANCE: (cont.)

2. Ambient QA Spikes: WHS personnel prepared the QA spikes since CAC QA was not available at that time. The beds of twenty resin tubes (SKC Lot # 499) were spiked with 70uL of 10 ng/uL Aldicarb (only) spike solution. This made a total of 700 ng/spike. The standard was secured from CAC Standards Repository. The Standard Solution # was 72-2922e. After spiking, the tubes were allowed to stand at ambient temperature for about one hour after which the broken ends of the primary sections were capped.

Five tubes were selected at random, extracted and analyzed for spiking level verification. Five additional tubes were retained in the lab in freezer # 27873 as Lab Spikes. The remaining 10 tubes were used by ARB staff as Trip Spikes and Field Spikes. When the tubes were returned to the lab, all 15 tubes were extracted and analyzed concurrently. The following table lists the % recoveries.

**TABLE 4. AMBIENT QA SPIKES - % RECOVERY**

CAC Spike ID	ARB Spike ID	Amount Spiked ng	% Recovery Aldicarb	% Recovery Sulfoxide	% Recovery Sulfone	% Recovery Total Aldicarb
QA-LS-1	---	700	91.57	---	---	91.57
QA-LS-2	---	700	90.28	8.35	---	97.98*
QA-LS-3	---	700	90.57	7.70	---	97.67*
QA-LS-4	---	700	95.57	---	---	95.57
QA-LS-5	---	700	93.86	9.07	---	102.2*
QA-TS-1	BAK-T1	700	80.14	8.16	---	87.67*
QA-TS-2	BAK-T2	700	83.43	10.52	---	93.13*
QA-TS-3	BAK-T3	700	85.00	8.87	---	93.18*
QA-TS-4	BAK-T4	700	90.57	7.48	---	97.47*
QA-TS-5	BAK-T5	700	74.14	10.44	---	83.77*
QA-FS-1	BAK-8S1	700	12.43	86.00	20.71	109.5*
QA-FS-2	BAK-8S2	700	---	77.57	20.71	89.28*
QA-FS-3	BAK-9S3	700	---	88.00	21.57	99.63*
QA-FS-4	BAK-9S4	700	---	18.42	---	17.00**
QA-FS-5	BAK-10S5	700	18.28	71.71	14.42	96.76*

\* Total Aldicarb recoveries include Aldicarb metabolite amounts that were corrected for molecular weights.

\*\* This result was double checked and verified as reported.

Please see attachment F1 ➤ F3 for resin tube Lab, Trip and Field Spike chromatograms.

## 5. QUALITY CONTROL:

A. Collection efficiencies and storage stability: For collection efficiencies and storage stability data, please refer to Attachment B for the Method SOP as developed by WHS.

B. Resin sample/extract integrity: Once received in the lab, all of the resin samples and spikes were stored in Freezer # 27873. The temperature of this freezer is recorded manually every work day. The average temperature of this freezer during the storage of samples and spikes was -16 ° C. At no time did the temperature vary more than +/- 3 ° C.

In all cases, the resin samples and spikes were analyzed on the same day that they were extracted.

C. On-going Quality Control spikes: The following tables list the WHS Laboratory on-going QC spike recoveries. For each set of 20 samples analyzed, one resin tube was spiked with either 15 ng or 1000 ng of Aldicarb (only). These levels covered the dynamic range of the LC detector and the recoveries are listed in Table 5.

In addition, for each set of 20 samples analyzed, a second resin tube was spiked with 50 ng each of Aldicarb, SO and SO<sub>2</sub>. The recoveries are listed in Table 6.

**TABLE 5. WHS LABORATORY ON-GOING QC SPIKES -% RECOVERY ALDICARB ONLY**

Date Analyzed	Lab ID	Sample ID	Amount Spiked ng	% Recovery Aldicarb
4-16-97	Spike #1 - A	Resin spike	15	76.6
4-17-97	Spike #1 - B	Resin spike	15	85.3
4-28-97	Spike #1 - C	Resin spike	15	105
4-29-97	Spike #1 - D	Resin spike	15	88.0
4-30-97	Spike #1 - E	Resin spike	15	66.7 *
5-5-97	Spike #1 - F	Resin spike	15	78.7
5-6-97	Spike #1 - G	Resin spike	15	99.3
6-23-97	Spike #1 - H	Resin Spike	1000	100.3
6-30-97	Spike #1 - I	Resin Spike	1000	79.7
7-7-97	Spike #1 - J	Resin Spike	1000	98.2

\* This low recovery appears to have been related to a problem with the test tube calibration.

## 5. QUALITY CONTROL: (cont.)

**TABLE 6. WHS LABORATORY ON-GOING QC SPIKES -% RECOVERY AT 50 ng EACH**

Date Analyzed	Lab ID	Sample ID	% Recovery Aldicarb	% Recovery Aldicarb SO	% Recovery Aldicarb SO <sub>2</sub>
4-16-97	Spike #2 - A	Resin Spike	80.6	80.0	87.4
4-17-97	Spike #2 - B	Resin Spike	85.2	99.8	97.2
4-28-97	Spike #2 - C	Resin Spike	86.4	94.4	84.4
4-29-97	Spike #2 - D	Resin Spike	94.6	92.6	108
4-30-97	Spike #2 - E	Resin Spike	97.6	105	115
5-5-97	Spike #2 - F	Resin Spike	84.2	102	90.8
5-6-97	Spike #2 - G	Resin Spike	87.2	122	104
6-23-97	Spike #2 - H	Resin Spike	106.0	126.0	114
6-30-97	Spike #2 - I	Resin Spike	82.5	91.3	82.5
7-7-97	Spike #2 - J	Resin Spike	86.8	116.0	95.2

Please see Attachment G1 ➤ G2 for a resin spike chromatograms.

D. On-going Quality Control resin blanks: The following table lists the resin blank results that were analyzed as part of the WHS Laboratory on-going QC for this Aldicarb study. Please see Attachment H for a chromatogram of a resin blank sample.

**TABLE 7. WHS LABORATORY ON-GOING QC RESIN BLANK RESULTS**

Date Analyzed	Lab ID	Sample ID	Aldicarb, SO and SO <sub>2</sub>
4-16-97	Blank A	Resin Blank	ND
4-17-97	Blank B	Resin Blank	ND
4-28-97	Blank C	Resin Blank	ND
4-29-97	Blank D	Resin Blank	ND
4-30-97	Blank E	Resin Blank	ND
5-5-97	Blank F	Resin Blank	ND
5-6-97	Blank G	Resin Blank	ND
6-23-97	Blank H	Resin Blank	ND
6-30-97	Blank I	Resin Blank	ND
7-7-97	Blank J	Resin Blank	ND

## 6. DISCUSSION:

Please see Attachment I for a chromatogram of an ARB Aldicarb resin sample.



**ATTACHMENT A-1**

**CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812**

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date: 1/1  
Sample/Run #: Days 1, 2  
Job name: \_\_\_\_\_  
Log numbers: 1-10

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or dry ice
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	3/28/97	1324	L.J.	S. Mayfield	Dry ICE
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
<u>1</u>	<u>SJ-1</u>	
<u>2</u>	<u>HEL-1</u>	
<u>3</u>	<u>FP-1</u>	
<u>4</u>	<u>BOB-1</u>	
<u>5</u>	<u>ARB-1</u>	
<u>6</u>	<u>SJ-2</u>	
<u>7</u>	<u>HEL-2</u>	
<u>8</u>	<u>FP-2</u>	
<u>9</u>	<u>BOB-2</u>	
<u>10</u>	<u>ARB-2</u>	

*Samples stored in freezer # 27873. Sm 3-28-97*

ATTACHMENT A-2

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date: 1 / 1  
Sample/Run #: DAY 3  
Job name: \_\_\_\_\_  
Log numbers: 11-20

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or dry ice
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	3/28/97	1324	J.J.	S. MARGHEB	DRY ICE
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
11	ST-3	
12	ST-3D	DUPLICATE
13	HEL-3	
14	HEL-3D	DUPLICATE
15	FP-3	
16	FP-3D	DUPLICATE
17	BOR-3	
18	BOR-3D	DUPLICATE
19	ARB-3	
20	ARB-3D	DUPLICATE

*Samples stored in freezer #27873 Sm 3-28-97*

# ATTACHMENT A-3

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

## ALDICARB AMBIENT CHAIN OF CUSTODY

### SAMPLE RECORD

Job #: C97-004

Date: 1/1  
Sample/Run #: BLANK, DAY 4  
Job name: \_\_\_\_\_  
Log numbers: 21-26

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or dry ice
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	3/28/97	1324	J.S.	J. Maughan	DRY ICE
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
21	B-3	BLANK DONE AT ARB SITE
22	SJ-4	
23	HEL-4	
24	FP-4	
25	BOR-4	
26	ARB-4	

*Samples stored in freezer # 27873. Sm 3-28-97*

ATTACHMENT A-4

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

**ALDICARB APPLICATION  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-007

Date: 3/28/97  
Sample/Run #: \_\_\_\_\_  
Job name: Aldicarb Application  
Log numbers: 1-20

ACTION			INITIALS <i>KEH</i>		METHOD OF STORAGE freezer, ice or <u>dry ice</u>
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	<i>3/25/97</i>	<i>1345</i>	<i>KEH</i>	<i>J. Maugh</i>	
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
<u>1</u>	<u>ALB W1</u>	
<u>2</u>	<u>" N1</u>	
<u>3</u>	<u>" E1</u>	
<u>4</u>	<u>" E10</u>	
<u>5</u>	<u>" S1</u>	
<u>6</u>	<u>" B1</u>	
<u>7</u>	<u>" S2</u>	
<u>8</u>	<u>" E2</u>	
<u>9</u>	<u>" E20</u>	
<u>10</u>	<u>" N2</u>	
<u>11</u>	<u>" W2</u>	
<u>12</u>	<u>" B2</u>	
<u>13</u>	<u>" S3</u>	
<u>14</u>	<u>" E3</u>	
<u>15</u>	<u>" E30</u>	
<u>16</u>	<u>" N3</u>	
<u>17</u>	<u>" W3</u>	
<u>18</u>	<u>" B3</u>	
<u>19</u>	<u>FS154</u>	
<u>20</u>	<u>ALB S4</u>	

Samples stored in freezer #27873. Sm 3-28-97

**ATTACHMENT A-5**

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

**ALDICARB APPLICATION  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-007

Date: 3/28/97  
Sample/Run #: \_\_\_\_\_  
Job name: Aldicarb Application  
Log numbers: 21 - 38

ACTION			INITIALS <i>KEM</i>		METHOD OF STORAGE freezer, ice or <u>dry ice</u>
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	<i>3/28/97</i>	<i>13 45</i>	<i>KEM</i>	<i>J. Mung</i>	
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
21	ALD E4	
22	" F4D	
23	FS2 N4	
24	ALD N4	
25	FS3 W4	
26	ALD W4/	
27	FS4 B4	
28	ALD B4	
29	TS1	
30	TS2	
31	TS3	
32	TS4	
33	ALD S5	
34	" F5	
35	" E5D	
36	" N5	
37	" W5	
38	" B5	
39		

*Samples stored in freezer #27873. Sm 3-28-97*

**ATTACHMENT A-6**

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date: 1 / 1  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 27-36

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or <u>dry ice</u>
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	3-4-97		LDT	CB	
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
27	SJ-5-	
28	HEL-5-	
29	FP-5-	
30	BOR-5-	
31	ARB-5-	
32	SJ-6-	
33	HEL-6-	
34	FP-6-	
35	BOR-6-	
36	ARB-6-	

Stored in freezer 27873. CB 4-4-97 *Carolina  
Lomson*

# ATTACHMENT A-7

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

## ALDICARB AMBIENT CHAIN OF CUSTODY

### SAMPLE RECORD

Job #: C97-004

Date: 1 1  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 37-46

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or <u>dry ice</u>
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	3-4-97		LDT	CB	
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
37	SS-7	
38	SS-7D	
39	HEL-7	
40	HEL-7D	
41	FP-7	
42	FP-7D	
43	BOR-7	
44	BOR-7D	
45	ARB-7	
46	ARB-7D	

Stored in freezer 27873. Carolinda Benson 4-4-97

**ATTACHMENT A-8**

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date:   /  /    
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers:   47-52  

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or <u>dry ice</u>
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	4-4-97		LOT	CB	
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
47	BLANK	
48	SJ-8-	
49	HEL-8-	
50	FP-8-	
51	BOR-8-	
52	PRB-8-	

Stored in freezer 27873. Carolinda Benson 4-4-97



**ATTACHMENT A-9**

**CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812**

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date:   /  /    
Sample/Run #:                       
Job name:                       
Log numbers:   53-62  

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or <u>dry ice</u>
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	4/11/97		<i>[Signature]</i>	<i>[Signature]</i>	
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
116	53	SS-9
127	54	HEL-9
128	55	FP-9
129	56	BOR-9
130	57	ARB-9
131	58	SS-10
132	59	HEL-10
133	60	FP-10
134	61	BOR-10
135	62	ARB-10

Stored in 27873

**ATTACHMENT A-10**

**CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812**

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date: 1 / 1  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 63-72

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or dry ice
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	4/11/97		DGP	M/W/ol	
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
63	SS-11	
64	SS-11D	
65	HBL-11	
66	HBL-11D	
67	FP-11	
68	FP-11D	
69	BOR-11	
70	BOR-11D	
71	ARB-11	
72	ARB-11D	

WHSC

136

137

138

139

140

141

142

143

144

145

Stored in 27973

**ATTACHMENT A-11**

**CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812**

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date:   /  /    
Sample/Run #:                       
Job name:                       
Log numbers:   73-78  

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or <u>dry ice</u>
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	4/11/97		DPH	M/Wal	
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
73	SS-12	
74	HBL-12	
75	FP-12	
76	BOR-12	
77	ARB-12	
78	Blank	

VASC  
 146  
 147  
 148  
 149  
 150  
 151

Stored in 27873

**ATTACHMENT A-12**

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date: 6/13/97  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 79-88

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or <u>dry ice</u>
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	6/13	1630	LOT	SSW	
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
79	WAS1	
80	MCF	
81	MAP1	
82	SEV1	
83	BAK1	
84	WAS2	
85	WAS2D	
86	MCF2	
87	MCF2D	
88	MAP2	

*Samples stored in #27837 Sm 6-13-97  
#27873 Sm 6-13-97 (Wing)*

**ATTACHMENT A-13**

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date: 6/13/  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 89-99

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or <u>(dry ice)</u>
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	6/13	1630	LDT	SSW	
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
89	MAP20	
90	SEV 2	
91	SEV20	
92	BAR 2	
93	BAR22	
94	BLNK	
95	WAS3	
96	MCF3	
97	MAP3	
98	SEV3	
99	BAR3	

*Samples stored in #27837 Sm 6-13-97  
#27873 Sm 6-13-97 (wrong entry)*

**ATTACHMENT A-14**

**CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812**

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date: 6/20/97  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 100-109

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or <u>dry ice</u>
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	6/20/97	1430	EXL RJ	C. Benson	dry ice
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
✓ 100	MCF 4	
✓ 101	WAS 4	
✓ 102	MAP 4	
✓ 103	SEV 4	
✓ 104	BAK 4	
✓ 105	MCF 5	
✓ 106	WAS 5	
✓ 107	MAP 5	
✓ 108	SEV 5	
✓ 109	BAK 5	

into freezer # 27873

# ATTACHMENT A-15

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

## ALDICARB AMBIENT CHAIN OF CUSTODY

### SAMPLE RECORD

Job #: C97-004

Date: 6/20/97  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 110 - 119

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or dry ice
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	6/20/97	1430	EXL RJ	C. Benson	dry ice
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
✓ 110	MCFL6	
✓ 111	MCFL6-D	Dup.
✓ 112	WAS6	
✓ 113	WAS6-D	Dup.
✓ 114	MAP6	
✓ 115	MAP6-D	Dup.
✓ 116	SEV6	
✓ 117	SEV6-D	Dup.
✓ 118	BAK6	
✓ 119	BAK6-D	Dup.

into freezer # 27873 48

**ATTACHMENT A-16**

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date: 6/20/97  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 120-125

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or dry ice
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	6/20/97	0 1430	EXL RA	C. Benson	dry ice
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
✓ 120	MCF 7	
✓ 121	WAS 7	
✓ 122	MAP 7	
✓ 123	SEV 7	
✓ 124	BAK 7	
✓ 125	Blank 1	

into freezer #27873



**ATTACHMENT A-17**

**CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812**

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date: 1 1  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 126-135

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or dry ice
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	6-27-97	3:59pm	LDT	S. Mangalath	✓
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
126	MCF-8.	
127	WAS-8.	
128	MAZ-8.	
129	SEV-8.	
130	BAA-8.	
131	BAK-8S1.	
132	BAK-8S2.	
133	BAK-T1.	
134	BAK-T2.	
135	BAK-T3.	

*Stored in freezer #27873 on 6-27-97*

**ATTACHMENT A-18**

**CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812**

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date: 1 / 1  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 136-145

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or <u>dry ice</u>
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	6-27-97	3:59pm	LDT	S. Mangione	✓
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
136	BAK-4.	
137	BAK-5.	
138	MCF-9.	
139	MPS-9.	
140	MAP-9.	
141	SEV-9.	
142	BAK-9.	
143	BAK-93.	
144	BAK-954.	
145	MCF-10.	

*stand in freezer #27873 Sm 6-27-97*

ATTACHMENT A-19

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date: 1/1  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 146-155

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or dry ice
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	6-27-97	3:59 <sub>pm</sub>	LDT	S. Margulies	✓
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
146	MCF-100	
147	WAS-100	
148	WAS-100	
149	MAP-100	
150	MAP-100	
151	SEV-100	
152	SEV-100	
153	BAX-100	
154	BAX-100	
155	BLANK	

*Stored in freezer # 27873. Sm 6-27-97*

**ATTACHMENT A-20**

**CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812**

**ALDICARB AMBIENT  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-004

Date: 1/1  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 156-161

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or dry ice
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	6-27-97	3:59pm	LOT	S. Mang. Tull	dry ice
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
156	BAK-1055	
157	MLF-11.	
158	WAS-11.	
159	MAP-11.	
160	SEV-11.	
161	BAK-11.	

*stored in freezer # 27873 Sm 6-27-97*

California Department of Food and Agriculture  
Center for Analytical Chemistry  
Worker Health & Safety Laboratory  
3292 Meadowview Road  
Sacramento, CA 95832  
916-262-2079

Original Date: 06-11-97  
Supersedes: New  
Page: 1 of 9

Standard Operating Procedure for the Analysis of  
Aldicarb and Metabolites on XAD-2 Resin Air Sampling Tubes

1. SCOPE:

This is an HPLC post column fluorescence detector method for the determination of Aldicarb, Aldicarb Sulfoxide, and Aldicarb Sulfone on XAD-2 resin air sampling tubes.

2. SUMMARY OF METHOD:

The exposed XAD-2 resin tubes are stored on dry ice or in the freezer until extraction. Samples are equilibrated to room temperature, desorbed with 4 mLs of methanol, and reduced in volume 2:1 prior to injection. The validated injection volume is 100  $\mu$ L, comprised of 30  $\mu$ L of standard or sample and 70  $\mu$ L of water. Depending on system performance, up to 40  $\mu$ L of sample or standard may be injected. The samples are analyzed by HPLC with autosampler, a C18 column, post column system, OPA derivatization, and fluorescence detection.

3. EQUIPMENT AND CONDITIONS:

A. Instrumentation:

Hewlett Packard 1050 Liquid Chromatograph with Autosampler  
Fluorescence Detector: SP Thermo Separation Products Spectra System  
FL2000  
Pickering Post Column System PCX5100  
Column: Pickering Laboratories Carbamate Analysis Column, 5 $\mu$ , C18,  
4.6 x 250 mm  
Data System: Hewlett Packard 3396 Series II Integrator

B. Conditions:

HPLC Pump Gradient: 10% ACN, hold for 2 min, T = 16 min 90% ACN,  
hold for 2 min, T = 20 min 10% ACN, ACN/H<sub>2</sub>O, 1 mL/min,  
Stoptime = 23 min, Posttime = 5 min.

B. Conditions: (cont.)

1050 ALS Injector Program: 1. Draw 30  $\mu$ L from Sample, Speed 200  $\mu$ L/min, Offset 0.0 mm. 2. Draw 70  $\mu$ L from Sample + 50, Speed 200  $\mu$ L/min, Offset 0.0 mm. 3. Mix max  $\mu$ L needle in seat, Repeat 10 times, Speed 500  $\mu$ L/min.

Post Column Parameters: Heated Reactor temperature = 100° C, Column temperature = 42° C, reagent preparation as per Pickering Laboratories PCX5100 User's Manual Version CE 1, January 1997.

Fluorescence detector: excitation at 330 nm, emission at 464 nm.

C. Auxiliary Apparatus:

1. Glass vials and caps, minimum 5 mL capacity
2. Vial Rotator, "Roto-Torque", Heavy Duty Rotator, Cole-Palmer Instrument Company, Chicago, IL
3. Nitrogen Evaporator, Organomation Associates Incorporated, Northborough, MA
4. Miscellaneous laboratory glassware

D. Reagents:

1. Methanol, acetonitrile, and water, HPLC grade
2. 0.05 M NaOH and *o*-phthalaldehyde reagent (OPA) as per Pickering Laboratories PCX5100 5100 User's Manual Version CE.1, January 1997.
3. Analytical standards of Aldicarb, Aldicarb Sulfoxide, and Aldicarb Sulfone in methanol
  - a) Stock standard of 1 mg/mL
  - b) Working standards in the range of 0.05 to 1 ng/ $\mu$ L

4. ANALYSIS OF SAMPLES:

1. Add 4 mLs of methanol to glass extraction vial.
2. Score the primary section end of the sampled XAD-2 tube above the retainer spring and break at the score. Remove the glass wool plug with forceps and place into the extraction vial. Pour the XAD-2 resin into vial. Retain the secondary section of the tube for later analysis if breakthrough is suspected.
3. Rotate or shake vial for 30 minutes.
4. Remove an aliquot of methanol extract and reduce 2:1 under nitrogen. Extract is ready for analysis. If the sample is outside the linear range of the standard curve, concentrate or dilute as necessary.

## 5. QUALITY ASSURANCE:

### A. Instrument Linearity and Reproducibility:

Instrument linearity and reproducibility were first evaluated by running 10 replicate 30  $\mu\text{L}$  injections of three levels of standards containing Aldicarb, Aldicarb Sulfoxide (SO), and Aldicarb Sulfone ( $\text{SO}_2$ ) over a 13 hour period. Please see Attachment 1 > 3 for linearity and reproducibility spreadsheets of Aldicarb, Aldicarb SO and Aldicarb  $\text{SO}_2$ . Please see Attachment 4 > 6 for graphs of r-values.

For sample analysis, standard curve linearity and reproducibility are tracked during each analytical run. For a 4 point standard curve ranging from 0.05  $\text{ng}/\mu\text{L}$  to 1.0  $\text{ng}/\mu\text{L}$  linearity is acceptable if  $r \geq 0.995$  and if instrument response for a given level is within  $\pm 20\%$  of its calculated value.

Instrument reproducibility is acceptable if responses for a given standard curve fall within  $\pm 20\%$  of the preceding curve.

### B. Limits of Detection and Quantitation:

The Limit of Detection (LOD) for Aldicarb was based on at least a 3:1 signal to noise ratio\*\* and an average peak height of 7 mm for the lowest standard of 0.05  $\text{ng}/\mu\text{L}$ . Calculation is as follows:

$$\frac{\text{LOD peak height}}{\text{standard peak height}} \times \text{ng standard injected} \times \frac{\text{final volume}}{\mu\text{L sample injected}} = \mu\text{g/sample}$$

For example:

$$\frac{3 \text{ mm}}{7 \text{ mm}} \times 1.5 \text{ ng standard injected} \times \frac{2 \text{ mL final volume}}{30 \mu\text{L injected}} = 0.043 \mu\text{g/sample}$$

The Limit of Quantitation (LOQ),\*\* based on ten times the signal to noise to ratio, or 3.33 x the LOD, is:

$$3.33 \times .043 = 0.143 \text{ or } 0.140 \mu\text{g/sample}$$

As per the above calculation, the LODs for Aldicarb Sulfoxide and Sulfone were based on average peak heights of 5 mm and 6 mm, respectively. This results in an LOD of 0.060  $\mu\text{g}/\text{sample}$  and an LOQ of 0.200  $\mu\text{g}/\text{sample}$  for the Sulfoxide and an LOD of 0.050  $\mu\text{g}/\text{sample}$  and an LOQ of 0.165  $\mu\text{g}/\text{sample}$  for the Sulfone.

B. Limits of Detection and Quantitation: (cont.)

For actual sample analysis, the total micrograms of the three analytes may be reported.

Results are reported as micrograms per sample. Calculation is:

$$\mu\text{g/sample} = \frac{(\text{sample peak height})(\mu\text{L of std injected})(\text{sample final volume, in mLs})}{(\text{std peak height})(\mu\text{L of sample injected})}$$

\*\* See Standard Operating Procedure WHS-QA-1, revision #1.

C. Extraction and Collection Efficiencies:

The following table lists the extraction efficiencies of Aldicarb (only) at the method LOQ, 2 x LOQ and 5 x LOQ from unseasoned XAD tubes.

**TABLE 1. EXTRACTION EFFICIENCIES OF UNSEASONED XAD TUBES**

Level	Spike ID	Amount spiked ng	Amount Recovered ng	% Recovery
1 x LOQ	Rep #1	140.0	121.7	86.93
1 x LOQ	Rep #2	140.0	117.0	83.57
1 x LOQ	Rep #3	140.0	111.0	79.29
1 x LOQ	Rep #4	140.0	105.6	75.43
1 x LOQ	Rep #5	140.0	108.0	77.14
				AVG.: 80.47
2 x LOQ	Rep #1	280.0	235.1	83.96
2 x LOQ	Rep #2	280.0	262.8	93.86
2 x LOQ	Rep #3	280.0	222.0	79.29
2 x LOQ	Rep #4	280.0	Lab Accident	Lab Accident
2 x LOQ	Rep #5	280.0	235.2	84.00
				AVG.: 85.28
5 x LOQ	Rep #1	700.0	638.3	91.19
5 x LOQ	Rep #2	700.0	582.6	83.23
5 x LOQ	Rep #3	700.0	637.3	91.04
5 x LOQ	Rep #4	700.0	684.0	97.71
5 x LOQ	Rep #5	700.0	615.0	87.86
				AVG.: 90.21



C. Extraction and Collection Efficiencies: (cont.)

The following table lists the collection and extraction efficiencies of Aldicarb (only) at the method LOQ, 2 x LOQ and 5 x LOQ from seasoned XAD tubes. The tubes were spiked and then subjected to an air flow of 4L/min for 24 hours at an ambient temperature of about 85 ° F before the primary section was extracted and analyzed.

**TABLE 2. COLLECTION AND EXTRACTION EFFICIENCIES OF SEASONED XAD TUBES**

Level	Spike ID	Amount spiked ng	Amount Recovered ng	% Recovery
1 x LOQ	Rep #1	140.0	141.7	101.21
1 x LOQ	Rep #2	140.0	125.1	89.36
1 x LOQ	Rep #3	140.0	144.2	103.00
1 x LOQ	Rep #4	140.0	169.8	121.29
1 x LOQ	Rep #5	140.0	126.3	90.21
				AVG.: 101.0
2 x LOQ	Rep #1	280.0	331.1	118.25
2 x LOQ	Rep #2	280.0	311.3	111.18
2 x LOQ	Rep #3	280.0	290.3	103.68
2 x LOQ	Rep #4	280.0	380.2	135.79*
2 x LOQ	Rep #5	280.0	332.7	118.82
				AVG.: 113.0
5 x LOQ	Rep #1	700.0	697.7	99.67
5 x LOQ	Rep #2	700.0	Lab Accident	Lab Accident
5 x LOQ	Rep #3	700.0	735.3	105.04
5 x LOQ	Rep #4	700.0	683.2	97.60
5 x LOQ	Rep #5	700.0	567.9	81.13
				AVG.: 95.86

\* This data point was not used to compute the average. It is believed to have been the result of a calibration problem.

## D. Storage Stability:

Two sets of 15 resin tube storage stability spikes were done. One set was spiked with 50 ng/spl of Aldicarb. The other set was spiked with 100 ng/spl of Sulfoxide and Sulfone (each). All tubes were spiked at the same time. They were unseasoned per the study director. One set of tubes was extracted and analyzed immediately (Time 0), the remainder of the tubes were placed in freezer #27873.

Spikes were analyzed in triplicate at Time 0 = extracted immediate spiking, at Time 1 = day 8, at Time 2 = day 15, at Time 3 = day 22, and at Time 4 = day 30. Time 4 extracts were rerun on day 56 due to unacceptable metabolite integration on day 30. The following tables (3,4,5) list the storage spike recoveries.

TABLE 3. STORAGE STABILITY SPIKES - 50 ng/spl of Aldicarb

Time	Spike ID	ng Spiked	ng Recovered	% Recovery	Mean	S. D.	% CV
Day 0	SS Spike #1	50.0	44.8	89.60			
	SS Spike #2	50.0	41.7	83.40			
	SS Spike #3	50.0	43.6	87.20	86.73	3.13	3.61
Day 8	SS Spike #1	50.0	52.7	105.4			
	SS Spike #2	50.0	48.0	96.00			
	SS Spike #3	50.0	47.5	95.00	98.80	5.74	5.81
Day 15	SS Spike #1	50.0	44.5	89.00			
	SS Spike #2	50.0	51.1	102.2			
	SS Spike #3	50.0	43.3	86.60	92.60	8.40	9.07
Day 22	SS Spike #1	50.0	37.4 *	74.80			
	SS Spike #2	50.0	36.7	73.40			
	SS Spike #3	50.0	54.4 **	108.8	85.67	20.05	23.40
Day 56	SS Spike #1	50.0	53.1	106.2			
	SS Spike #2	50.0	46.5	93.00			
	SS Spike #3	50.0	42.4	84.80	94.67	10.80	11.41

\* This spike had 9.9 ng/spl of Aldicarb Sulfoxide. Total Recovery = 94.60%

\*\* This spike had 6.3 ng/spl of Aldicarb Sulfoxide. Total Recovery = 121.40 %

Original Date: 06-11-97

Supersedes: New

Page: 7 of 9

D. Storage Stability: (cont.)

**TABLE 4. STORAGE STABILITY SPIKES - 100 ng/spl of Aldicarb SO**

Time	Spike ID	ng Spiked	ng Recovered	% Recovery	Mean	S. D.	% CV
Day 0	SS Spike #1	100.0	110.7	110.7			
	SS Spike #2	100.0	102.9	102.9			
	SS Spike #3	100.0	87.6	87.60	100.4	11.75	11.70
Day 8	SS Spike #1	100.0	100.1	100.1			
	SS Spike #2	100.0	96.1	96.10			
	SS Spike #3	100.0	132.1	132.1	109.43	19.73	18.03
Day 15	SS Spike #1	100.0	104.9	104.9			
	SS Spike #2	100.0	103.5	103.5			
	SS Spike #3	100.0	88.7	88.70	99.03	8.98	9.07
Day 22	SS Spike #1	100.0	103.4	103.4			
	SS Spike #2	100.0	123.8	123.8			
	SS Spike #3	100.0	88.9	88.90	105.37	17.53	16.64
Day 56	SS Spike #1	100.0	94.9	94.90			
	SS Spike #2	100.0	95.0	95.0			
	SS Spike #3	100.0	113.3	113.3	101.07	10.59	10.48

D. Storage Stability: (cont.)

**TABLE 5. STORAGE STABILITY SPIKES - 100 ng/spl of Aldicarb SO<sub>2</sub>**

Time	Spike ID	ng Spiked	ng Recovered	% Recovery	Mean	S. D.	% CV
Day 0	SS Spike #1	100.0	109.3	109.3			
	SS Spike #2	100.0	102.6	102.6			
	SS Spike #3	100.0	97.1	97.10	103.00	6.11	5.93
Day 8	SS Spike #1	100.0	106.2	106.2			
	SS Spike #2	100.0	103.0	103.0			
	SS Spike #3	100.0	132.5	132.5	113.90	16.19	14.21
Day 15	SS Spike #1	100.0	100.0	100.0			
	SS Spike #2	100.0	100.4	100.4			
	SS Spike #3	100.0	91.8	91.80	97.40	4.85	4.98
Day 22	SS Spike #1	100.0	105.5	105.5			
	SS Spike #2	100.0	116.7	116.7			
	SS Spike #3	100.0	109.2	109.2	110.47	5.71	5.17
Day 56	SS Spike #1	100.0	94.6	94.60			
	SS Spike #2	100.0	94.5	94.50			
	SS Spike #3	100.0	105.0	105.0	98.03	6.03	6.15

**6. DISCUSSION:**

Seasoned spiking shows that Aldicarb metabolizes to its Sulfoxide and Sulfone under actual sample conditions. Therefore, the three analytes are included in all QC measures.

The validation of this method included spiking at the LOQ, 2 x LOQ, and 5 x LOQ. Breakthrough was not observed at any level.

The Hewlett Packard 1050 Autosampler Injector Program was used to combine and mix 30 µL of standard or sample with 70 µL of water before injection. This way, the solvent composition of the sample more closely approximates the mobile phase, allowing larger injection volumes and greater sensitivity. Although no mixing takes place when the combined injection volumes equal the capacity of the sample loop, peak shape is substantially improved.

Original Date: 06-11-97  
Supersedes: New  
Page: 9 of 9

Written By:

*Carolinda Benson*

Carolinda Benson, Agricultural Chemist  
Worker Health & Safety Laboratory

Approved By:

*Sheila Margetich*

Sheila Margetich, Supervisor  
Worker Health & Safety Laboratory

Reviewed By:

*for Valuk For Terry Jackson*

Terry Jackson, QA Officer  
Center for Analytical Chemistry

Project: ARB Aldicarb and Metabolites on XAD-2 Resin Air Sampling Tubes  
 Chromatogram I.D.: ARB-6b 3-21,22-97 WHS-QA-2 #3  
 Instrument: 1050LC/PCX 5100 spl30/70H2O LC/Mth1 C18 25cm ex330em464 aldica40.dat

# ATTACHMENT I

## Aldicarb Sulfone

	Conc	rt	Area	Int	Calx10-5*	Lin**	Repro***	
Curve 1	0.05	7.669	58766	vb	2.552			
r = 0.99997	0.1	7.661	102599	pb	2.924	87.29%		
	1	7.640	1049174	bb	2.859	102.26%		
Curve 2	0.05	7.645	63130	pb	2.376		107.43%	
r = 0.99996	0.1	7.635	106073	pb	2.828	84.01%	103.39%	
	1	7.635	1060317	pb	2.829	99.96%	101.06%	
Curve 3	0.05	7.642	55070	vb	2.722		87.23%	
r = 0.99998	0.1	7.640	115010	pb	2.608	104.42%	108.43%	
	1	7.645	1063625	bb	2.821	92.48%	100.31%	
Curve 4	0.05	7.640	68200	pb	2.199		123.84%	
r = 0.99992	0.1	7.640	103390	vb	2.902	75.80%	89.90%	
	1	7.638	1056363	bb	2.840	102.17%	99.32%	
Curve 5	0.05	7.635	56417	vb	2.659		82.72%	
r = 1.0000	0.1	7.630	111472	vb	2.691	98.79%	107.82%	
	1	7.641	1079407	pb	2.779	96.83%	102.18%	
Curve 6	0.05	7.639	54675	pb	2.743		96.91%	
r = 0.99999	0.1	7.629	114632	pb	2.617	104.83%	102.83%	
	1	7.641	1078054	pb	2.783	94.04%	99.87%	
Curve 7	0.05	7.638	50547	pb	2.968		92.45%	
r = 0.99997	0.1	7.625	113937	pb	2.633	112.70%	99.39%	
	1	7.647	1087786	pb	2.758	95.47%	100.90%	
Curve 8	0.05	7.649	59330	vb	1.528		117.38%	
r = 1.0000	0.1	7.634	112712	pb	2.662	94.99%	98.92%	
	1	7.640	1081926	bb	2.773	95.99%	99.46%	
Curve 9	0.05	7.643	51669	pb	2.903		87.09%	
r = 0.99985	0.1	7.650	126309	vb	2.375	122.23%	112.06%	
	1	7.643	1093214	pb	2.744	86.55%	101.04%	
Curve 10	0.05	7.639	64566	vb	2.323		124.96%	
r = 1.0000	0.1	7.642	118106	vb	2.540	91.46%	93.51%	
	1	7.637	1096886	pb	2.735	92.87%	100.34%	
							Repro with Curve 1	Repro with Curve 5
							109.87%	114.44%
							115.11%	105.95%
							104.55%	101.62%

ARB.WB10

\*Calibration Factor: Calculated by HP 3396 Series II Integrator. Is equal to ngs of standard divided by peak area.

\*\*Linearity: Calculated by Quattro Pro. Sample calculation : (peak area for .1ng/ul standard divided by 2 x peak area for .05ng/ul standard) x 100

\*\*\* Reproducibility: Calculated by Quattro Pro. Sample calculation : (peak area for .05ng/ul concentration of 2nd standard curve divided by peak area for .05ng/ul concentration for 1st standard curve) x 100

Project: ARB Aldicarb and Metabolites on XAD-2 Resin Air Sampling Tubes  
 Chromatogram I.D.: ARB-6b 3-21,22-97 WHS-QA-2 #3  
 Instrument: 1050LC/PCX 5100 spl30/70H2O LC/Mth1 C18 25cm ex330em464 aldica40.dat

# ATTACHMENT 2

## Aldicarb Sulfoxide

	Conc	rt	Area	Int	Calx10 <sup>-5</sup> *	Lin**	Repro***	
Curve 1	0.05	6.562	58368	vb	2.570			
r = 0.99995	0.1	6.553	94853	pb	3.163	81.25%		
	1	6.516	935982	pb	3.205	98.68%		
Curve 2	0.05	6.523	46782	pb	3.206		80.15%	
r = 0.99999	0.1	6.505	99710	pb	3.009	106.57%	105.12%	
	1	6.502	965815	pb	3.106	96.86%	103.19%	
Curve 3	0.05	6.505	55070	vb	2.724		117.72%	
r = 0.99998	0.1	6.504	96408	pb	3.112	87.53%	96.69%	
	1	6.508	958915	pb	3.129	99.46%	99.29%	
Curve 4	0.05	6.515	34034	pb	4.407		61.80%	
r = 0.99983	0.1	6.5069	101524	pb	2.995	149.15%	105.31%	
	1	6.502	964603	pb	3.110	95.01%	100.59%	
Curve 5	0.05	6.501	45917	pb	3.267		134.92%	
r = 1.0000	0.1	6.495	98090	pb	3.058	106.81%	96.62%	
	1	6.514	988366	bb	3.035	100.76%	102.46%	
Curve 6	0.05	6.503	51072	pb	2.937		111.23%	
r = 1.0000	0.1	6.493	100992	pb	2.971	98.87%	102.96%	
	1	6.506	960213	pb	3.124	95.08%	97.15%	
Curve 7	0.05	6.527	61743	pb	2.492		120.89%	
r = 0.99994	0.1	6.493	98534	pb	3.045	79.79%	97.57%	
	1	6.505	958507	pb	3.130	97.28%	99.82%	
Curve 8	0.05	6.522	57167	pb	2.624		92.59%	
r = 0.99992	0.1	6.486	93604	pb	3.205	81.87%	95.00%	
	1	6.512	992581	pb	3.022	106.04%	103.55%	
Curve 9	0.05	6.474	57140	vb	2.625		99.95%	
r = 1.0000	0.1	6.513	104939	pb	2.859	91.83%	112.11%	
	1	6.513	984122	pb	3.048	93.78%	99.15%	
Curve 10	0.05	6.522	71536	vb	2.097		125.19%	Repro with Curve 1
r = 0.99987	0.1	6.516	103611	pb	2.8985	72.42%	98.73%	Repro with Curve 5
	1	6.501	994820	pb	3.016	96.01%	101.09%	122.56%
								109.23%
								155.79%
								105.63%
								106.29%
								100.65%

\*Calibration Factor: Calculated by HP 3396 Series II Integrator. Is equal to ngs of standard divided by peak area.

\*\*Linearity: Calculated by Quattro Pro. Sample calculation : (peak area for .1ng/ul standard divided by 2 x peak area for .05ng/ul standard) x 100

\*\*\* Reproducibility: Calculated by Quattro Pro. Sample calculation : (peak area for .05ng/ul concentration of 2nd standard curve divided by peak area for .05ng/ul concentration for 1st standard curve) x 100

Aldicarb							
	Conc	rt	Area	Int	Cal x -5*	Lin**	Repro***
Curve 1	0.05	11.841	64765	pb	2.316		
r = 0.99999	0.1	11.846	130860	pb	2.293	101.03%	
	1	11.818	1235078	pb	2.429	94.38%	
Curve 2	0.05	11.836	58561	pb	2.561		90.42%
r = 0.99998	0.1	11.829	130259	pb	2.303	111.22%	99.54%
	1	11.824	1269031	pb	2.364	97.42%	102.75%
Curve 3	0.05	11.831	71750	pb	2.091		122.52%
r = 0.99995	0.1	11.824	121771	pb	2.464	84.86%	93.48%
	1	11.827	1274999	pb	2.353	104.70%	100.47%
Curve 4	0.05	11.828	76053	pb	1.972		106.00%
r = 0.99999	0.1	11.825	131398	pb	2.283	86.39%	107.91%
	1	11.824	1252398	pb	2.395	95.31%	98.23%
Curve 5	0.05	11.831	74189	pb	2.022		97.55%
r = 0.99999	0.1	11.816	132701	pb	2.261	89.43%	100.99%
	1	11.823	1308502	pb	2.293	98.61%	104.48%
Curve 6	0.05	11.825	75276	vb	1.993		101.47%
r = 0.99998	0.1	11.829	130805	vb	2.293	86.88%	98.57%
	1	11.836	1291564	pb	2.323	98.74%	98.71%
Curve 7	0.05	11.837	79700	pb	1.882		105.88%
r = 1.0000	0.1	11.829	141210	vb	2.124	88.59%	107.95%
	1	11.841	1279144	pb	2.345	90.58%	99.04%
Curve 8	0.05	11.840	62213	pb	2.411		78.06%
r = .99994	0.1	11.822	140277	pb	2.139	112.74%	99.34%
	1	11.821	1264511	pb	2.372	90.14%	98.86%
Curve 9	0.05	11.832	78348	vb	1.915		125.94%
r = 0.99999	0.1	11.843	137133	pb	2.188	87.52%	97.76%
	1	11.838	1297430	pb	2.312	94.61%	102.60%
Curve 10	0.05	11.826	73077	vb	2.053		93.27%
r = 0.99992	0.1	11.838	154440	pb	1.943	105.67%	112.62%
	1	11.833	1301573	pb	2.305	84.28%	100.32%
							Repro with Curve 1
							Repro with Curve 5
							112.83%
							98.50%
							118.02%
							116.38%
							105.38%
							99.47%

ARB.WS18

\*Calibration Factor: Calculated by HP 3396 Series II Integrator. Is equal to ngs of standard divided by peak area.

\*\*Linearity: Calculated by Quattro Pro. Sample calculation : (peak area for .1ng/ul standard divided by 2 x peak area for .05ng/ul standard) x 100

\*\*\* Reproducibility: Calculated by Quattro Pro. Sample calculation : (peak area for .05ng/ul concentration of 2nd standard curve divided by peak area for .05ng/ul concentration for 1st standard curve) x 100



## WHS LABORATORY METHOD VALIDATION FORM

PROJECT: ARBSTUDY DIRECTOR: Kevin MongarP.A.I.: Sheila MargetichSOP: WHS-QA-2, rev. #5, #3LAB BOOK: S96173

## Instrument Linearity Check

Instrument: Hewlett Packard 1050 HPLC S/N 3448A03614 with Pickering PCX 5100

Post Column System

Detector: Spectra Physics Fluorescence, em 330nm/ex 464nm

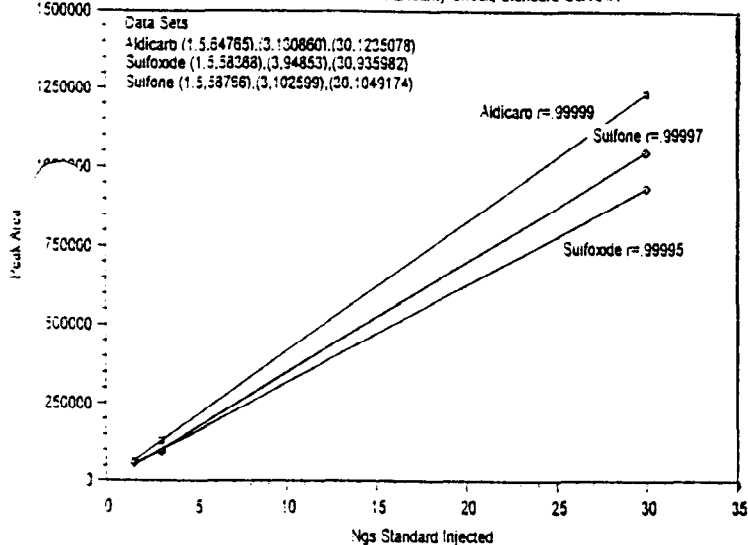
Analyte(s): Aldicarb, Aldicarb Sulfoxide, Aldicarb Sulfone standards in methanol

Chromatogram ID: ARB-6b

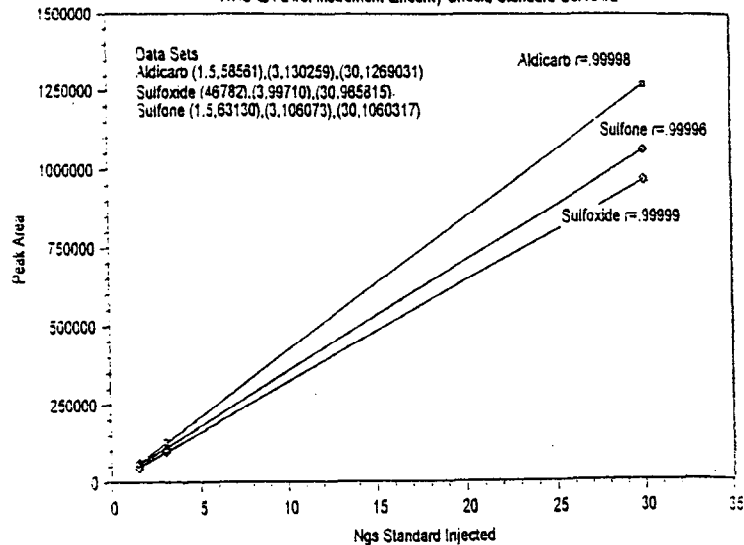
Date of Linearity Check: 3-21, 22-97

$$r = \frac{n \sum xy - \sum x \sum y}{\sqrt{\{n \sum x^2 - (\sum x)^2\} \{n \sum y^2 - (\sum y)^2\}}}$$

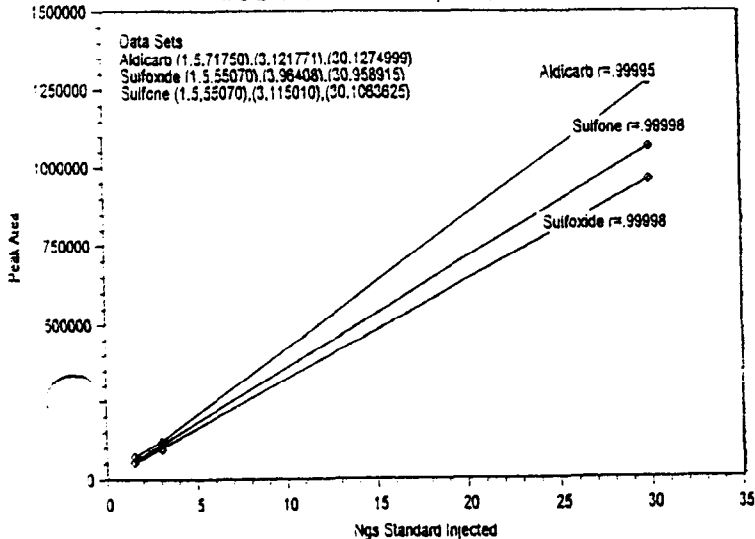
WHS-QA-2 #3: Instrument Linearity Check, Standard Curve #1



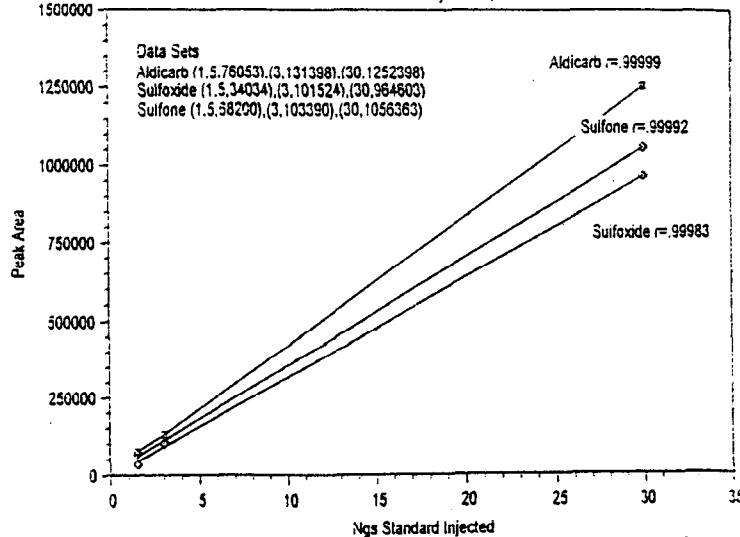
WHS-QA-2 #3: Instrument Linearity Check, Standard Curve #2



WHS-QA-2 #3: Instrument Linearity Check, Standard Curve #3



WHS-QA-2 #3: Instrument Linearity Check, Standard Curve #4



## WHS LABORATORY METHOD VALIDATION FORM

PROJECT: ARBSTUDY DIRECTOR: Kevin MongarP.A.I.: Sheila MargetichSOP: WHS-QA-2, rev. #5, #3LAB BOOK: S96173

## Instrument Linearity Check

Instrument: Hewlett Packard 1050 HPLC S/N 3448A03614 with Pickering PCX 5100  
Post Column System

Detector: Spectra Physics Fluorescence, em 330nm/ex 464nm

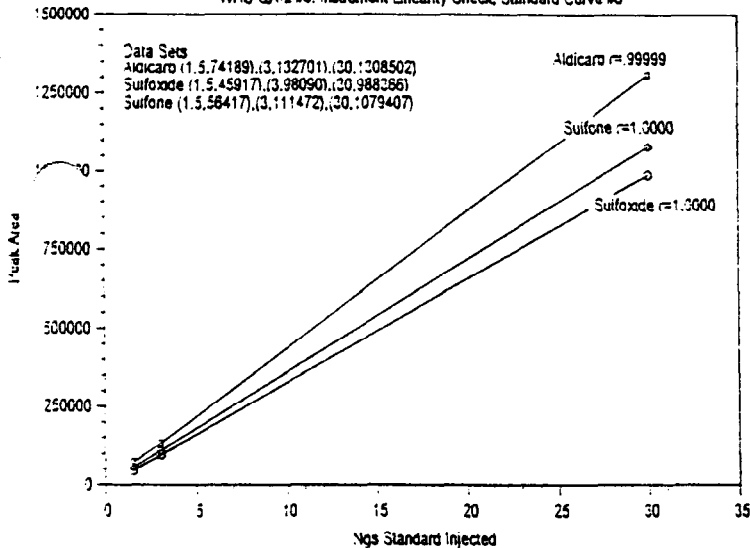
Analyte(s): Aldicarb, Aldicarb Sulfoxide, Aldicarb Sulfone standards in methanol

Chromatogram ID: ARB-6b

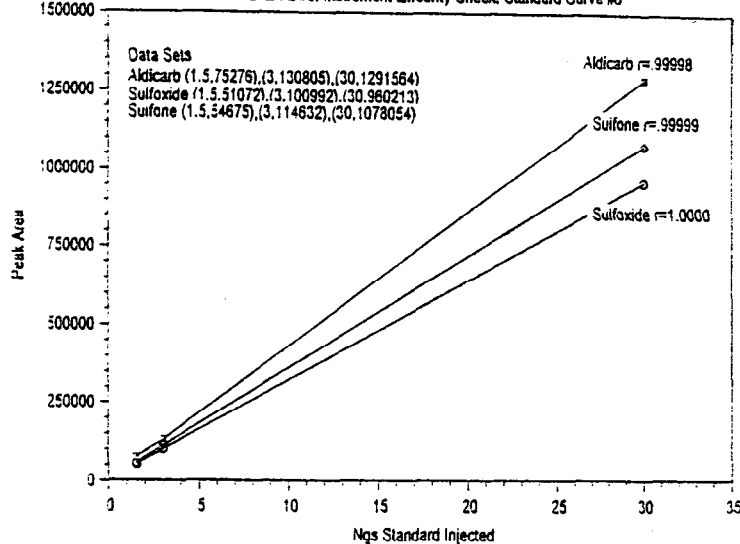
Date of Linearity Check: 3-21, 22-97

$$r = \frac{n \sum xy - \sum x \sum y}{\sqrt{\{n \sum x^2 - (\sum x)^2\} \{n \sum y^2 - (\sum y)^2\}}}$$

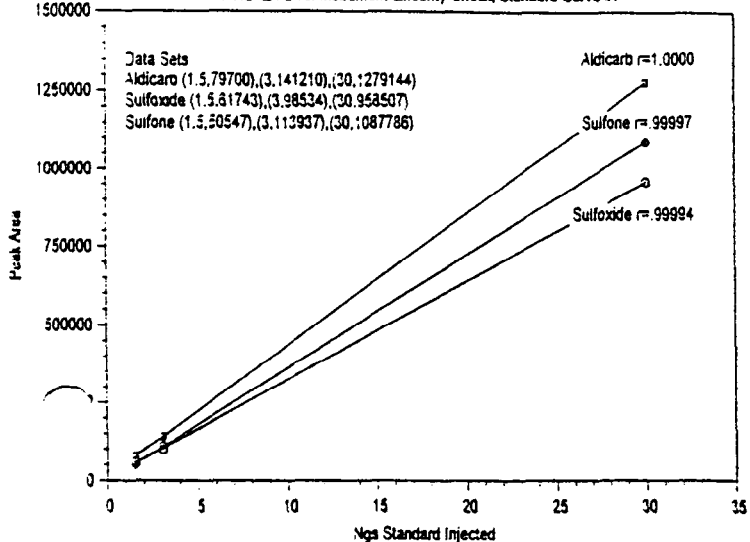
WHS-QA-2 #3: Instrument Linearity Check, Standard Curve #5



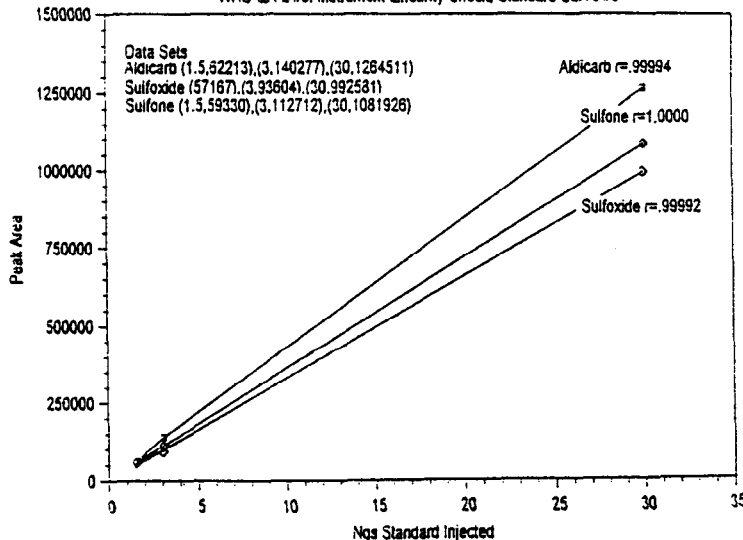
WHS-QA-2 #3: Instrument Linearity Check, Standard Curve #6



WHS-QA-2 #3: Instrument Linearity Check, Standard Curve #7



WHS-QA-2 #3: Instrument Linearity Check, Standard Curve #8



## WHS LABORATORY METHOD VALIDATION FORM

PROJECT: ARBSTUDY DIRECTOR: Kevin MongarP.A.I.: Sheila MargetichSOP: WHS-QA-2, rev. #5, #3LAB BOOK: S96173

## Instrument Linearity Check

Instrument: Hewlett Packard 1050 HPLC S/N 3448A03614 with Pickering PCX 5100  
Post Column System

Detector: Spectra Physics Fluorescence, em 330nm/ex 464nm

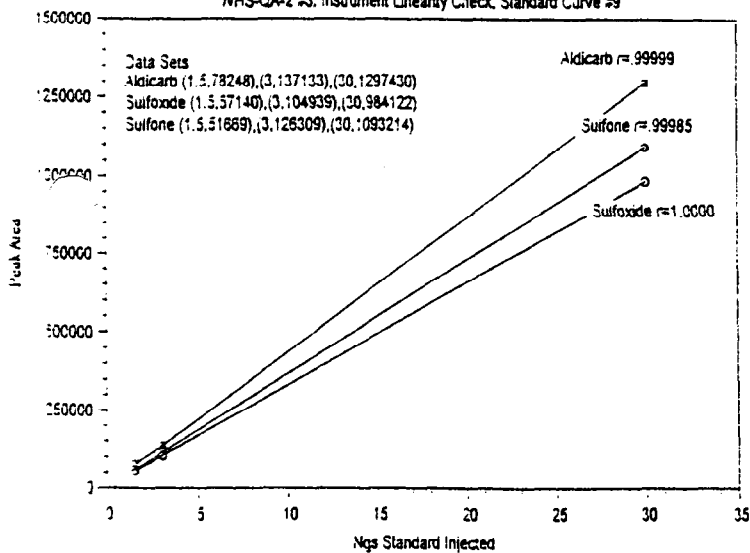
Analyte(s): Aldicarb, Aldicarb Sulfoxide, Aldicarb Sulfone standards in methanol

Chromatogram ID: ARB-6b

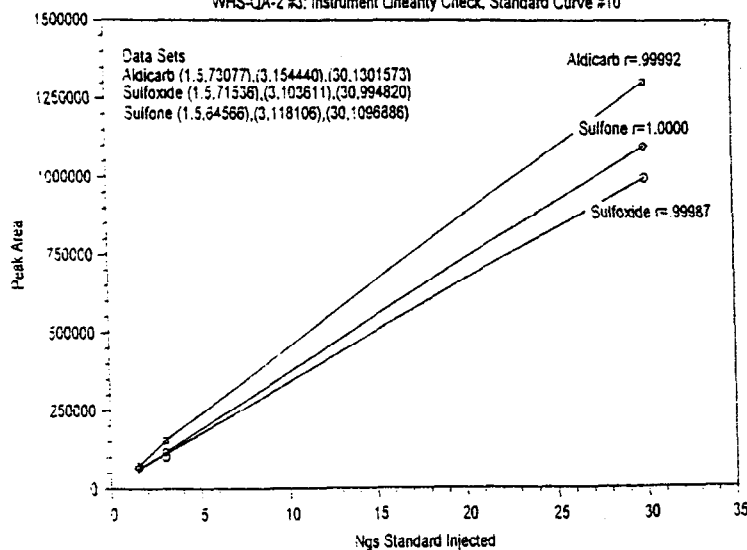
Date of Linearity Check: 3-21, 22-97

$$r = \frac{n \sum xy - \sum x \sum y}{\sqrt{\left[ n \sum x^2 - (\sum x)^2 \right] \left[ n \sum y^2 - (\sum y)^2 \right]}}$$

WHS-QA-2 #3: Instrument Linearity Check, Standard Curve #9



WHS-QA-2 #3: Instrument Linearity Check, Standard Curve #10



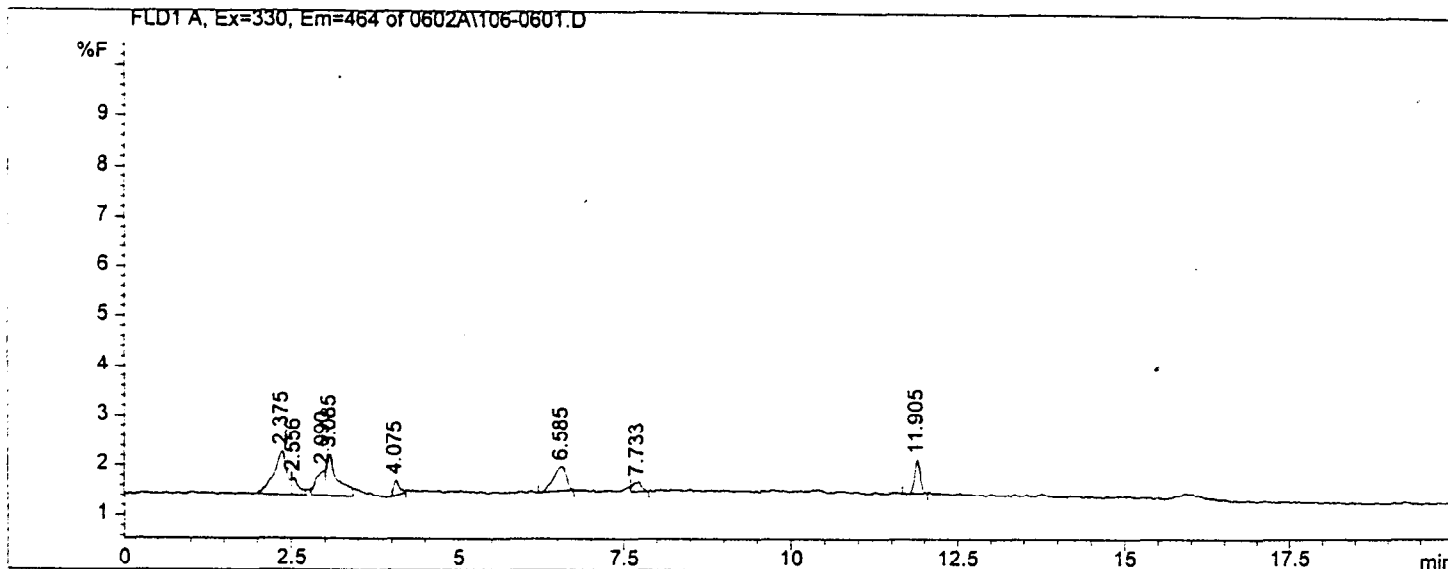
ATTACHMENT C  
Seasoned LOD Resin Spike - 50 ng/spl

ame: 50-2

=====  
Injection Date : 6/2/97 2:50:28 PM Seq. Line : 6  
Sample Name : 50-2 Vial : 106  
Acq. Operator : cb Inj : 1  
Inj Volume : Inj prog

Acq. Method : C:\HPCHEM\1\METHODS\CARB.M  
Last changed : 6/2/97 2:10:08 PM by cb  
Analysis Method : C:\HPCHEM\1\METHODS\CARB.M  
Last changed : 6/3/97 10:51:10 AM by cb  
(modified after loading)

=====  
Instrument Conditions: At Start At Stop  
Temperature: 24.3 24.4 °C  
Pressure: 165.2 165.4 bar  
Flow: 1.000 1.000 ml/min



## External Standard Report

Sorted by Retention Time

Calib. Data Modified : Tuesday, June 03, 1997 10:44:19 AM  
Multiplier : 1.667000e-2  
Dilution : 1.000000  
Uncalibrated Peaks : compound name not specified

Signal 1: FLD1 A, Ex=330, Em=464

Injection Date : 6/2/97 2:50:28 PM

Seq. Line : 6

Sample Name : 50-2

Vial : 106

Acq. Operator : cb

Inj : 1

Inj Volume : Inj prog

Acq. Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 6/2/97 2:10:08 PM by cb

Analysis Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 6/3/97 10:51:10 AM by cb

(modified after loading)

RT [min]	Sig	Type	Area [%F*s]	Amt/Area	Amount [ug/spl]	Grp	Name
2.375	1	VV	12.04306	0.00000	0.00000	?	
2.556	1	VV	2.56568	0.00000	0.00000	?	
2.990	1	VV	4.56051	0.00000	0.00000	?	
3.085	1	VV	9.73752	0.00000	0.00000	?	
4.075	1	PV	1.90652	0.00000	0.00000	?	
6.585	1	PV	6.61181	2.97628e-1	3.28043e-2		sulfoxide
7.733	1	VV	1.79836	3.02531e-1	9.06950e-3		sulfone
11.905	1	PV	4.14061	2.18828e-1	1.51043e-2		aldicarb
22.581	1	VV	2.62287	0.00000	0.00000	?	
22.667	1	VV	1.00735	0.00000	0.00000	?	

Totals :

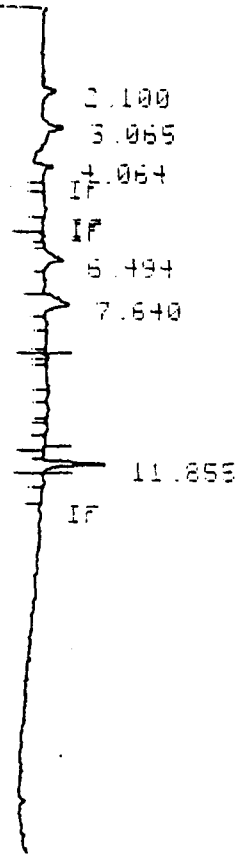
5.69781e-2

\*\*\* End of Report \*\*\*

$1.9 \times 10^{-2} \times 10 \text{ ng std} \times \frac{100 \text{ mL}}{40 \mu\text{L}}$   
 $= .00548 \text{ ug/spl sulfone}$   
 CB 6-30-97  
 6-3-97

BT  
RL  
BT

ATTACHMENT D-1  
Standard Curve Chromatogram - 0.025 ng/ul



STOP

---AMOUNT UNITS ARE MOG/SPL---

HP 1050 LC SPN 3448A03614

AUTOSAMPLER INJECTION APR 17, 1997 06:08:08

SAMPLE # : ID CODE :

1 0.025 NG/UL

ALDICARB 50 502 CARBON 25CM 10-10/1-90/10-90/10-10/20 ACN/H2O 1ML/MIN  
40UL CB

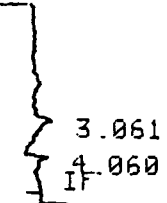
ESTD

RT	HEIGHT	AREA	WIDTH	TYPE	UGS	NAME
2.100	2021	25213	.208	UU		
3.065	3166	53689	.309	UU		
4.064	2240	14928	.111	PV		
6.494	2307	35004	.253	UB	1.0000	SULFOXIDE
106.771 % OF OLD CF VALUE						
7.640	2336	22957	.164	BB	1.0000	SULFONE
191.192 % OF OLD CF VALUE						
11.855	7339	50825	.115	PB	1.0000	ALDICARB
99.4609 % OF OLD CF VALUE						
RECALIBRATING LEVEL-- 1						
AREA CALIB						

START PENDING

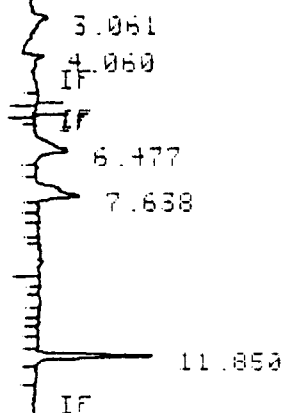
RUN # 29 APR 17, 1997 06:37:47

START



3745  
3746  
3747

ATTACHMENT D-2  
Standard Curve Chromatogram - 0.050 ng/ul



STOP

---AMOUNT UNITS ARE MCG/SPL---

HP 1050 LC 5/1N 3448A03614

AUTOSAMPLER INJECTION APR 17, 1997 06:37:47

SAMPLE # : ID CODE :

2 0.05 NG/UL

ALDICARB 50 502 CARBODOL 25CM 10-10/1-90/16-90/13-10/20 ACN/H2O 1ML/MIN  
40UL CB

ESTD

RT	HEIGHT	AREA	WIDTH	TYPE	UGS	NAME
3.061	2652	51697	.325	UU		
4.060	2483	16681	.125	PU		
6.477	3941	70370	.300	PB	2.0000	SULFOXIDE
99.7573 % OF OLD CF VALUE						
7.638	5129	77702	.252	PB	2.0000	SULFONE
93.5381 % OF OLD CF VALUE						
11.850	13370	93014	.116	UB	2.0000	ALDICARB
96.007 % OF OLD CF VALUE						

RECALIBRATING LEVEL-- 2

AREA CALIB

START PENDING

RUN # 30 APR 17, 1997 07:07:23

START

2.077

3.061

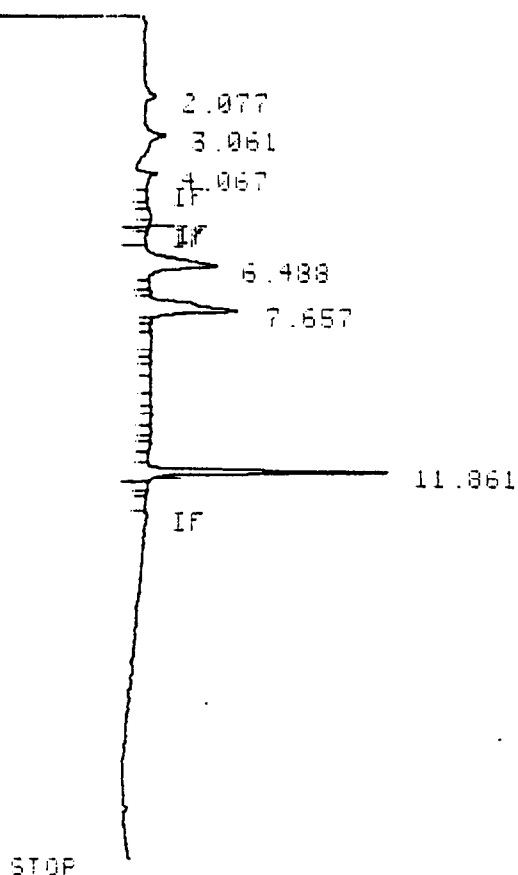
4.067

IF

IF

**ATTACHMENT D-3**  
**Standard Curve Chromatogram - 0.10 ng/ul**

START  
 RUN # 30 APR 17, 1997 07:07:23  
 START



---AMOUNT UNITS ARE MG/SPL---

HP 1050 LC S/N 3448A03614

AUTOSAMPLER INJECTION APR 17, 1997 07:07:23

SAMPLE # : ID CODE :

3 .1 NG/UL

ALDICARB 50 502 CARBOL 25CM 10-10/1-90/16-90/18-10/20 ACN/H2O 1ML/MIN  
 40UL 05

ESTD

RT	HEIGHT	AREA	WIDTH	TYPE	UGS	NAME
2.077	1832	36140	.329	UU		
3.061	3292	74353	.376	UP		
4.067	2399	18464	.128	PU		
6.488	8171	132120	.269	BB	4.0000	SULFOXIDE
96.0846 % OF OLD CF VALUE						
7.657	10191	140648	.230	UB	4.0000	SULFONE
103.326 % OF OLD CF VALUE						
11.861	27467	172590	.105	PB	4.0000	ALDICARB
98.5996 % OF OLD CF VALUE						

RECALIBRATING LEVEL-- 3

AREA CALIB

START PENDING

RUN # 31 APR 17, 1997 07:37:00

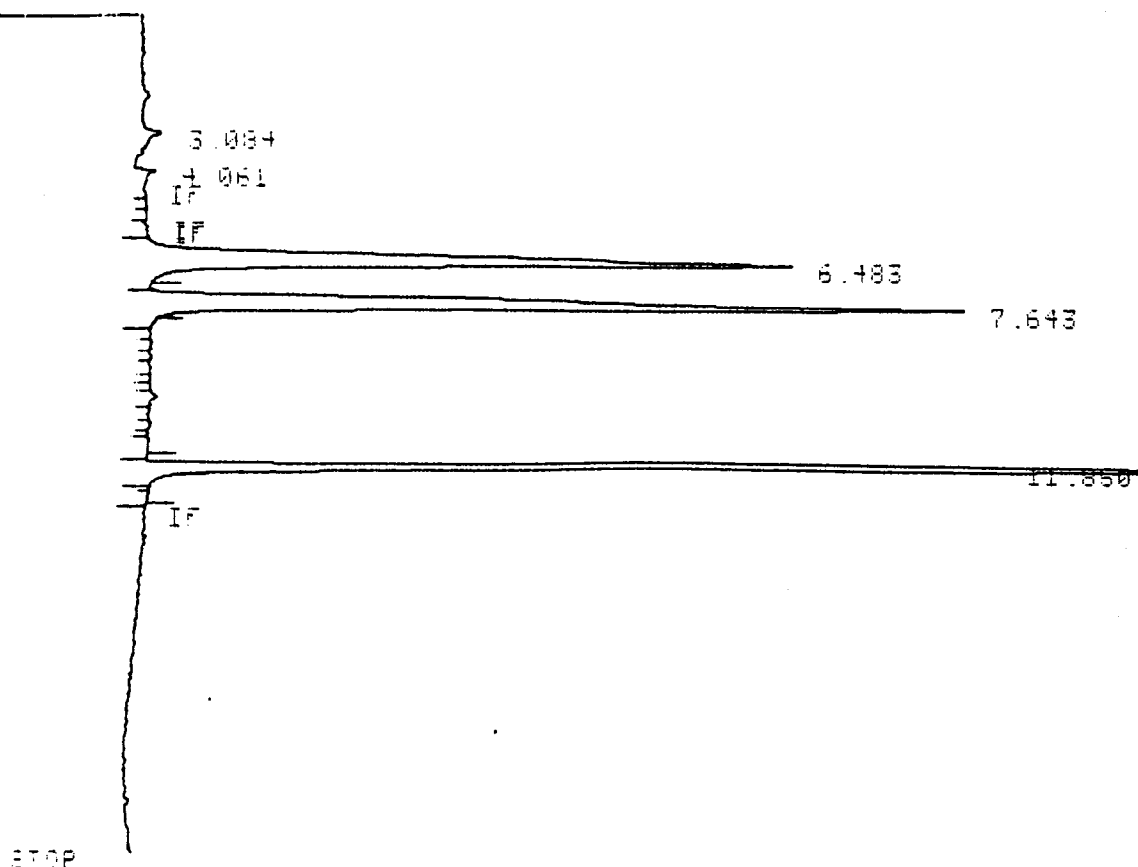
START

3.084



**ATTACHMENT D-4**  
**Standard Curve Chromatogram - 1.0 ng/ul**

PUN # 51 APR 17, 1997 07:37:00  
 START



---AMOUNT UNITS ARE MCG/SPL---

HP 1050 LC S/N 3448A03614

AUTOSAMPLER INJECTION APR 17, 1997 07:37:00

SAMPLE # : ID CODE :

4 1 NG/UL

ALDICARB 50 502 CARBOL 25CM 10-10/1-90/16-90/18-10/20 ACN/H2O 1ML/MIN  
 40UL CB

ESTD

RT	HEIGHT	AREA	WIDTH	TYPE	UGS	NAME
3.084	3043	57533	.315	UU		
4.061	2491	32596	.218	PV		
6.483	73665	1203535	.272	BB	40.0000	SULFOXIDE
101.647 % OF OLD CF VALUE						
7.643	93028	1361478	.244	BB	40.0000	SULFONE
101.063 % OF OLD CF VALUE						
11.850	265895	1732998	.109	PB	40.0000	ALDICARB
99.1112 % OF OLD CF VALUE						

RECALIBRATING LEVEL-- 4

AREA CALIB

== CURRENT CALIBRATION TABLE ==

ALDICARB 50 502 CARBOL 25CM 10-10/1-90/16-90/18-10/20 ACN/H2O 1ML/MIN  
 40UL CB

AREA CALIB

PEAK#	RT	WND	% AMT<NGS>	LEVEL	AREA/HT	CAL FACTOR	PEAK NAME
1	6.500	5.0	1.000	1	35004.00	2.857E-05	SULFOXIDE
			2.000	2	70870.02	2.822E-05	
			4.000	3	132120.00	3.028E-05	
			40.000	4	1203535.20	3.324E-05	

California Department of Food and Agriculture  
Center for Analytical Chemistry  
Worker Health and Safety Laboratory  
3292 Meadowview Road  
Sacramento, CA 95832

Number: WHS-AD-11  
Date: 02/05/96  
Revision:  
Replaces:  
Page: 1 of 3

## STANDARD OPERATING PROCEDURE

**Title:** Data Generation and Reporting

**Purpose:** To Provide a Standardized Procedure for the Generation and Reporting of Chromatographic Data

**Scope:** All laboratory personnel.

**Procedure:**

Any conflict with instructions in the method or protocol must be resolved with senior staff, the study director, and documented before proceeding.

The number of standards used should adequately describe the standard curve shape. Typically this is 3-5 points spanning 1-2 orders of magnitude for linear systems. For non-linear systems, additional points or narrower concentration ranges may be needed. Calibration curves should include a data point near the instrument MDL of the compound(s), or a point that approximates the project LOD. All samples with responses higher than the upper limit of the standard curve must be diluted and reanalyzed.

The number and concentration of standards necessary to "adequately describe" the curve shape depend on the type of curve fitting used for data analysis as well as the actual shape of the curve, which in turn depends on the detector used and the chemical being analyzed. In the case of point-to-point curve fitting (used by HP 5880 and 3396 integrators), the number of standards and their concentrations should be chosen so that the maximum quantitative error between a smooth curve and the point-to-point line, measured at the midpoint between consecutive standard levels, is 15% or less. Curve-fit errors in systems that can use quadratic functions (HP MSD, Varian Saturn) are much less, and consequently wider concentration ranges can be used.

In general, using peak heights for GC data will minimize errors because it reduces the effect of small leading or trailing peak interferences. For LC work, peak areas yield better data because of the tendency for LC peaks to widen and shorten during a run due to the effect of developing column voids.

Retention times should be reproducible to better than 1% in most cases for both LC and GC. Capillary GC and gradient LC times should be even better. Some systems will

slowly drift due to changing ambient conditions in the lab, but consecutive runs should show very small changes.

Samples must be run in groups small enough that the standard curves on either side of them will not vary by more than +/- 15%. Sufficient data should be generated during method development to provide guidance for the chemist on this number, and that information should be included in the method. Typically, no more than 10-20 samples should be analyzed between standard curves. 'Conditioning' samples and cooling GC analytical systems between batches may provide more consistent data.

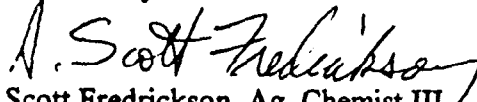
Residues are generally reported in micrograms/sample. In the absence of complicating factors, levels should be reported as follows:

>= 1000 ugs	to nearest 10 ug
100 to 999 ugs	to nearest ug
10 to 99.9 ugs	to nearest 0.1 ug
1 to 9.99 ugs	to nearest 0.01 ug
0.010 to .999 ugs	to nearest 0.001 ug

To prevent confusion when reporting high levels of residue, do not mix reporting units. That is, do not report some values as ugs/sample, and some as mgs/sample within the same group of samples, unless the unit changes are *clearly* marked to draw the reader's attention.

Recovery data should be reported, but sample results NOT corrected for recovery. If corrected results are reported, a notation explicitly stating that fact should be included on the report sheet.

Written By:

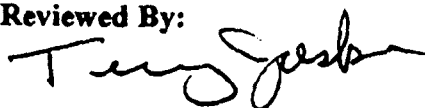
  
Scott Fredrickson, Ag. Chemist III  
Worker Health & Safety Laboratory

WHS-AD-11

Revision:

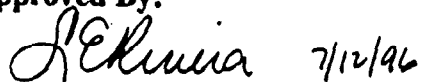
Page: 3 of 3

Reviewed By:



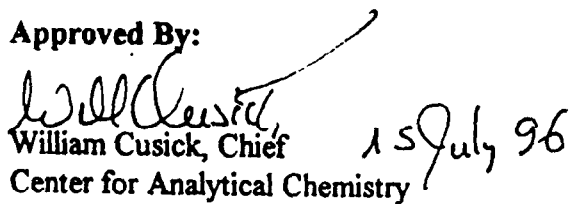
Terry Jackson, QA Officer  
Center for Analytical Chemistry

Approved By:



Lilia Rivera, Program Supervisor  
Center for Analytical Chemistry

Approved By:



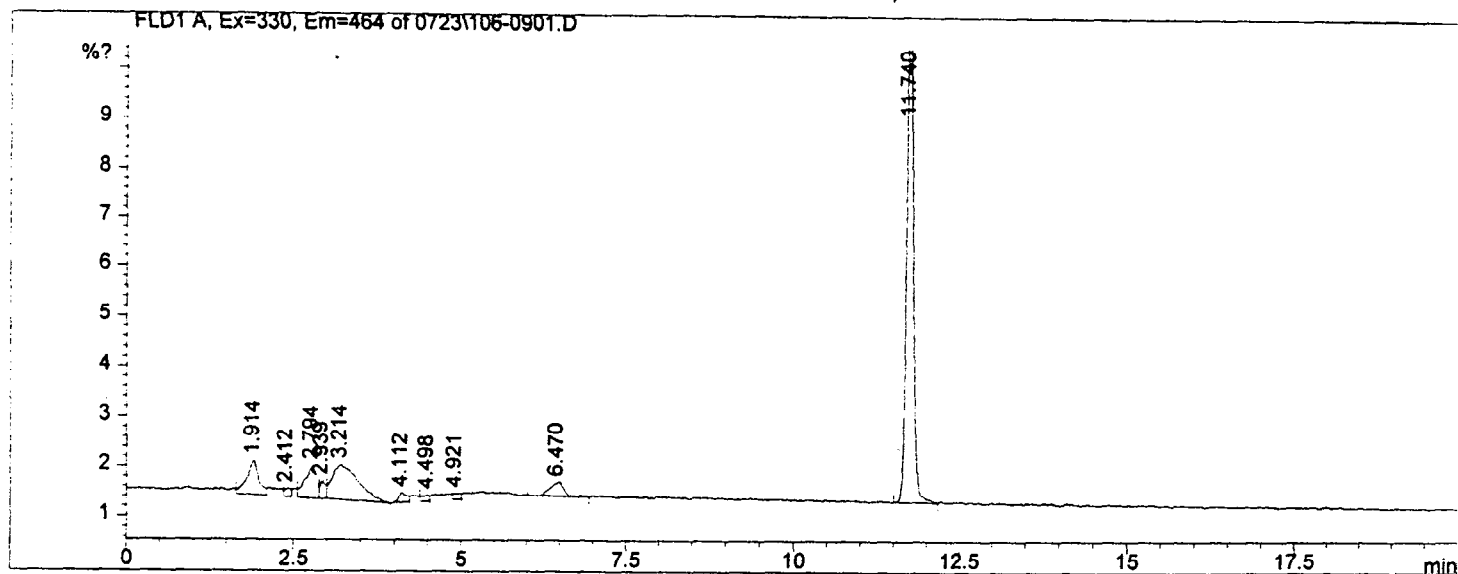
William Cusick, Chief  
Center for Analytical Chemistry

In:  
Sat:ATTACHMENT F-1  
Resin Lab Spike Chromatogram - 700 ng/splAcq. Operator : ~~sf~~ ③ CB 7-24-97  
CBInj : 1  
Inj Volume : Inj progSequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S  
Method : C:\HPCHEM\1\METHODS\CARB.M  
Last changed : 7/23/97 6:00:38 PM by ~~sf~~ CB  
(modified after loading)

carbamate method

Instrument Conditions: At Start At Stop

Temperature:	25.4	25.5	°C
Pressure:	155.4	153.8	bar
Flow:	1.000	1.000	ml/min



## External Standard Report

Sorted by Retention Time

Calib. Data Modified : Wednesday, July 23, 1997 5:33:23 PM  
Multiplier : 3.350000e-2  
Dilution : 1.000000  
Uncalibrated Peaks : compound name not specified

Signal 1: FLD1 A, Ex=330, Em=464

Injection Date : 7/23/97 6:04:09 PM

Seq. Line : 9

Sample Name : 51-QA-LS-2

Vial : 106

Acq. Operator : ~~sf~~

Inj : 1

CB ③ CB 7-24-97

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/23/97 6:00:38 PM by ~~sf~~ CB

(modified after loading)

carbamate method

RT [min]	Sig	Type	Area [%?s]	Amt/Area	Amount [ug/spl]	Grp	Name
1.914	1	VV	8.78569	0.00000	0.00000	?	
2.412	1	VV	1.19830	0.00000	0.00000	?	
2.794	1	VV	7.76844	0.00000	0.00000	?	
2.939	1	VV	2.10869	0.00000	0.00000	?	
3.214	1	VV	19.44749	0.00000	0.00000	?	
4.112	1	PV	1.55978	0.00000	0.00000	?	
4.498	1	VV	1.03624	0.00000	0.00000	?	
4.921	1	VV	1.04027	0.00000	0.00000	?	
6.470	1	BV	4.62190	3.78094e-1	5.85418e-2		sulfoxide
7.755	1	*	not found	*			sulfone
11.740	1	BV	72.43906	2.60319e-1	6.31718e-1		aldicarb
22.031	1	VV	2.42212	0.00000	0.00000	?	

Totals : 6.90259e-1

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

\*\*\* End of Report \*\*\*

CB 7-24-97

$$\frac{632 \text{ ng Aldicarb} + 58.5 \text{ ng Sulfoxide}}{700 \text{ ng Aldicarb spiked}} \times 100 = 98.64\%$$

=====

## ATTACHMENT F-2

Injec

Resin Trip Spike Chromatogram - 700 ng/spl

Sample Name

Vial : 112

Acq. Operator : *st* *CB* 7-24-97

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Method : C:\HPCHEM\1\METHODS\CARB.M

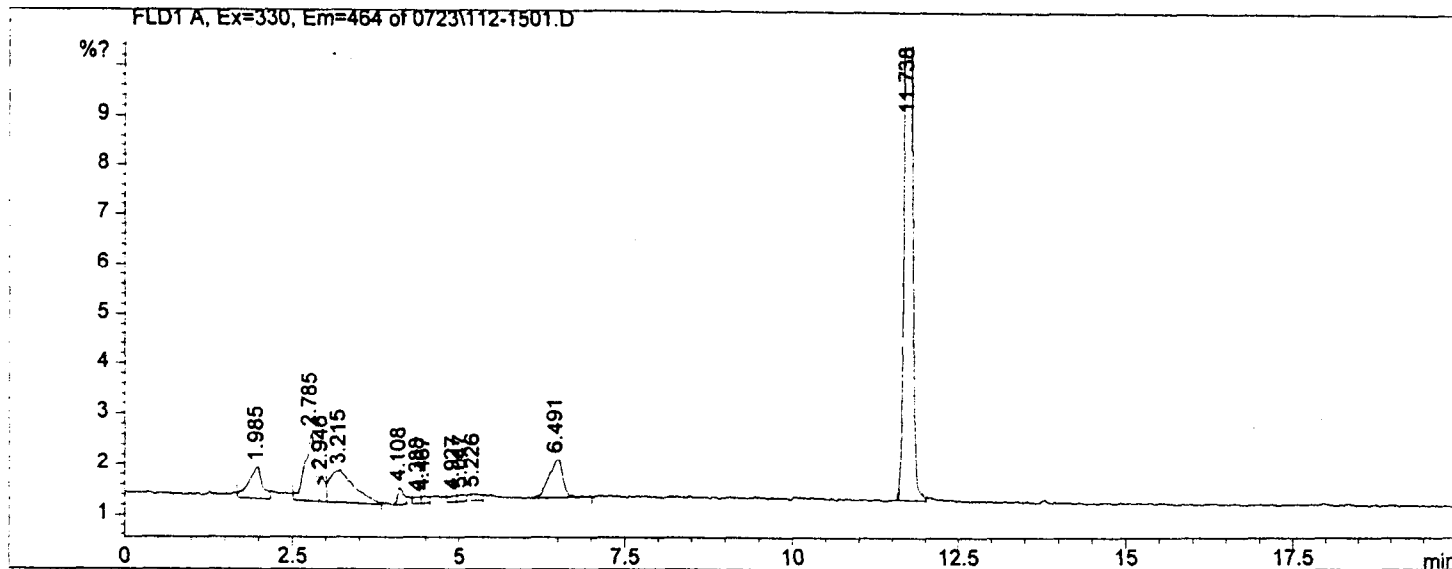
Last changed : 7/23/97 8:42:19 PM by *st* *CB*  
(modified after loading)

carbamate method

=====

Instrument Conditions: At Start At Stop

Temperature:	28.8	29.2	°C
Pressure:	145.5	147.4	bar
Flow:	1.000	1.000	ml/min



## External Standard Report

Sorted by Retention Time

Calib. Data Modified : Wednesday, July 23, 1997 5:33:23 PM

Multiplier : 1.667000e-2

Dilution : 1.000000

Uncalibrated Peaks : compound name not specified

Signal 1: FLD1 A, Ex=330, Em=464

Injection Date : 7/23/97 8:45:48 PM

Seq. Line : 15

Sample Name : 57-BAK-T3

Vial : 112

Acq. Operator : ~~sf~~ ③ CB 7-24-97

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/23/97 8:42:19 PM by ~~sf~~ CB

(modified after loading)

carbamate method

RT [min]	Sig	Type	Area [%?*s]	Amt/Area	Amount [ug/spl]	Grp	Name
1.985	1	VV	8.84629	0.00000	0.00000	?	
2.785	1	VV	14.15024	0.00000	0.00000	?	
2.946	1	VV	2.95972	0.00000	0.00000	?	
3.215	1	VV	16.04984	0.00000	0.00000	?	
4.108	1	PV	2.09543	0.00000	0.00000	?	
4.388	1	VV	1.02271	0.00000	0.00000	?	
4.467	1	VV	1.03121	0.00000	0.00000	?	
4.927	1	VV	1.08718	0.00000	0.00000	?	
5.047	1	VV	1.13125	0.00000	0.00000	?	
5.226	1	VV	1.39512	0.00000	0.00000	?	
6.491	1	PV	11.05078	3.37254e-1	6.21277e-2		sulfoxide
7.755	1	*	not found	*			sulfone
11.738	1	VV	137.16232	2.60263e-1	5.95091e-1		aldicarb
22.058	1	PV	1.87518	0.00000	0.00000	?	

Totals : 6.57218e-1

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

CB 7-24-97

\*\*\* End of Report \*\*\*

$$\frac{595 \text{ ng Aldicarb} + 62.1 \text{ ng Sulfoxide}}{700 \text{ ng Aldicarb spiked}} \times 100 = 93.87\%$$



Inject

## ATTACHMENT F-3

Sample Name

Resin Field Spike Chromatogram - 700 ng/spl

Acq. Operator : sf ③ CB 7-24-97

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

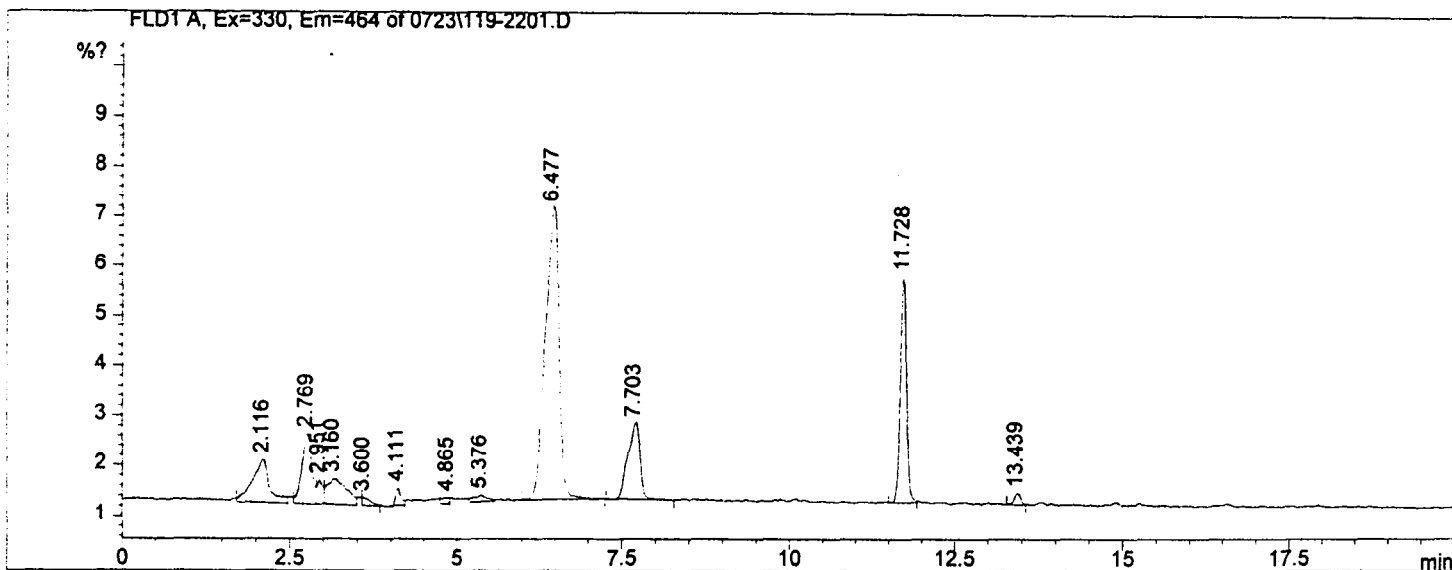
Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/23/97 11:50:53 PM by sf CB  
(modified after loading)

carbamate method

Instrument Conditions: At Start At Stop

Temperature:	30.8	30.9	°C
Pressure:	145.4	147.2	bar
Flow:	1.000	1.000	ml/min



## External Standard Report

Sorted by Retention Time

Calib. Data Modified : Wednesday, July 23, 1997 5:33:23 PM

Multiplier : 1.667000e-2

Dilution : 1.000000

Uncalibrated Peaks : compound name not specified

Signal 1: FLD1 A, Ex=330, Em=464

Injection Date : 7/23/97 11:54:18 PM

Seq. Line : 22

Sample Name : 64-BAK-10S5

Vial : 119

Acq. Operator : ~~sf~~ ③ CB 7-24-97  
CB

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/23/97 11:50:53 PM by ~~sf~~ CB  
(modified after loading)

carbamate method

RT [min]	Sig	Type	Area [%?*s]	Amt/Area	Amount [ug/spl]	Grp	Name
2.116	1	VV	14.94716	0.00000	0.00000	?	
2.769	1	VV	14.04611	0.00000	0.00000	?	
2.951	1	VV	2.84424	0.00000	0.00000	?	
3.160	1	VV	10.23351	0.00000	0.00000	?	
3.600	1	VV	1.44424	0.00000	0.00000	?	
4.111	1	PV	1.95796	0.00000	0.00000	?	
4.865	1	VV	1.03003	0.00000	0.00000	?	
5.376	1	VV	2.05381	0.00000	0.00000	?	
6.477	1	BBA	85.36217	3.52974e-1	5.02278e-1		sulfoxide
7.703	1	BV	19.32974	3.12501e-1	1.00696e-1		sulfone
11.728	1	BV	29.38222	2.60491e-1	1.27589e-1		aldicarb
13.439	1	VV	1.56218	0.00000	0.00000	?	
22.001	1	PV	1.21252	0.00000	0.00000	?	

Totals :

7.30563e-1

CB 7-24-97

\*\*\* End of Report \*\*\*

$$\frac{128 \text{ ng Aldicarb} + 502 \text{ ng Sulfoxide} + 101 \text{ ng Sulfone}}{700 \text{ ng Aldicarb spiked}} \times 100 = 104.43\%$$

4

## ATTACHMENT G-1

ES

## Resin On-Going QC Spike Chromatogram - 15 ng/spl Aldicarb

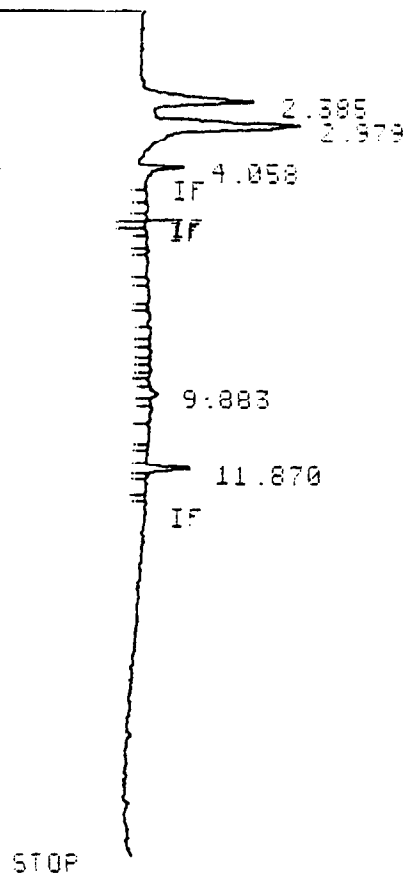
RT	HEIGHT	AREA	WIDTH	TYPE	UGS	NAME
2.385	14006	222989	.265	UU		
2.975	10416	195181	.312	UU		
4.050	3714	30806	.138	PU		

MULTIPLIER = 0.0166675  
 MLS OF SAMPLE = 0.6667

START PENDING

RUN # 26 APR 17, 1997 05:08:43

START



STOP

---AMOUNT UNITS ARE MCG/SPL---

HP 1050 LC S/N 3448A03614

AUTOSAMPLER INJECTION APR 17, 1997 05:08:43

SAMPLE # : ID CODE :

26

15NG ALD

ALDICARB 50 502 CARBOL 25CM 10-10/1-90/16-90/18-10/20 ACN/H2O 1ML/MIN  
40UL CB

ESTD

RT	HEIGHT	AREA	WIDTH	TYPE	UGS	NAME
2.385	13175	191733	.243	UU		
2.979	18375	341531	.310	UU		
4.058	5244	31953	.102	PU		
9.883	996	10767	.180	PB		
11.870	5151	34748	.112	PB	.0115	ALDICARB

MULTIPLIER = 0.0166675

MLS OF SAMPLE = 0.6667

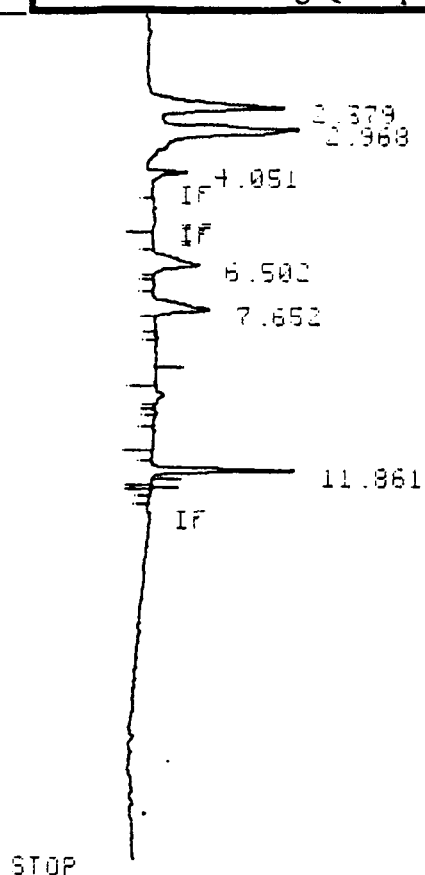
START PENDING

RUN # 27 APR 17, 1997 05:38:22

START PENDING  
RUN #  
START

# ATTACHMENT G-2

Resin On-Going QC Spike Chromatogram - 50 ng/spl Aldicarb, SO & SO<sub>2</sub> (each)



---AMOUNT UNITS ARE MCG/SPL---

HP 1050 LC S/N 3448A03614

AUTOSAMPLER INJECTION APR 17, 1997 05:38:22

SAMPLE # : ID CODE :

27 50NG SO SO2

ALDICARB SO SO2 CARRICOL 25CM 10-10/1-90/16-90/18-10/20 ACN/H2O 1ML/MIN  
400UL CB

ESTD

RT	HEIGHT	AREA	WIDTH	TYPE	UGS	NAME
2.379	15655	217804	.232	BU		
2.968	17437	315105	.301	UU		
4.051	4773	34784	.121	PU		
6.502	5399	92004	.253	PB	.0400	SULFOXIDE
7.652	6632	95161	.239	PB	.0437	SULFONE
11.861	16797	106284	.105	PB	.0403	ALDICARB

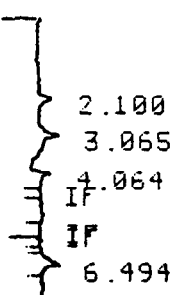
MULTIPLIER = 0.0186675

MLS OF SAMPLE = 0.6667

START PENDING

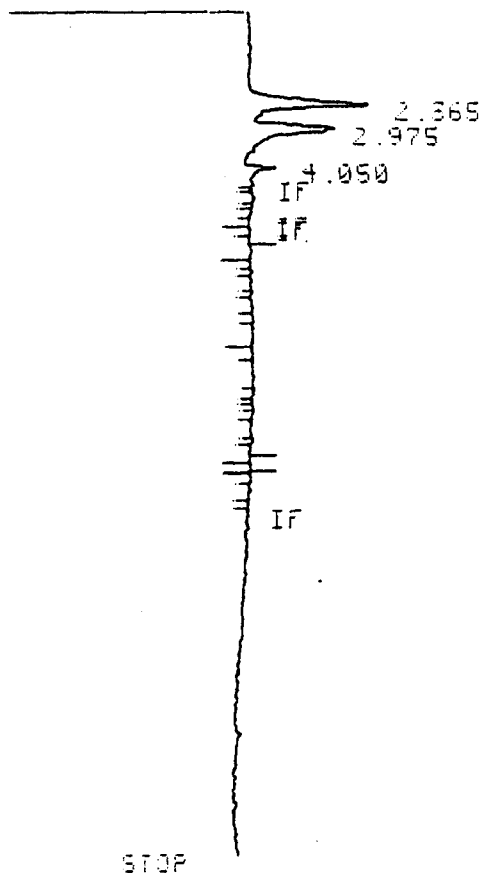
RUN # 28 APR 17, 1997 06:08:08

START



# **ATTACHMENT H** **Resin Blank On-Going QC Chromatogram**

START PENDING  
 RUN # 25 APR 17, 1997 04:39:01  
 START



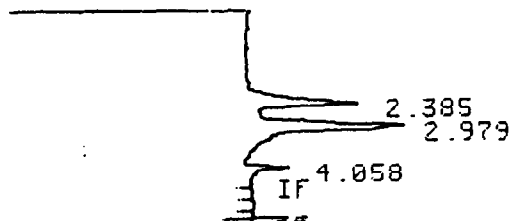
---AMOUNT UNITS ARE MCG/SPL---  
 HP 1050 LC S/N 3448A03614  
 AUTOSAMPLER INJECTION APR 17, 1997 04:39:01  
 SAMPLE # : 10 CODE :  
 25 LAB BLANK  
 ALDICARB 50 502 CARBOL 25CM 10-10/1-90/16-90/18-10/20 ACN/H2O 1ML/MIN  
 40UL CB

ESTD

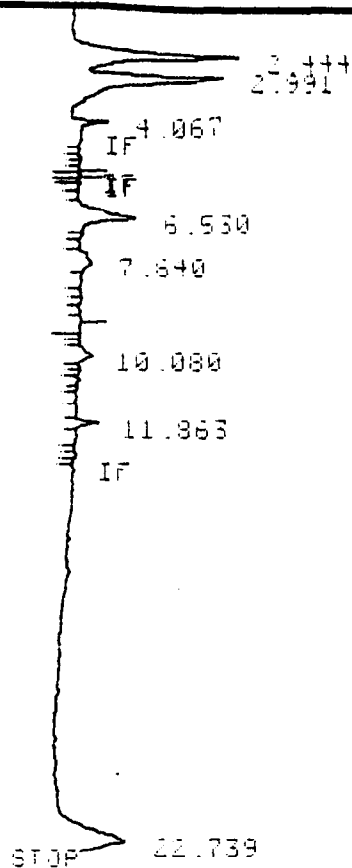
RT	HEIGHT	AREA	WIDTH	TYPE	UGS	NAME
2.365	14006	222989	.265	UU		
2.975	10416	195181	.312	UU		
4.050	3714	30806	.138	PU		

MULTIPLIER = 0.0166675  
 MLS OF SAMPLE = 0.6667

START PENDING  
 RUN # 26 APR 17, 1997 05:08:43  
 START



ATTACHMENT I  
ARB Resin Sample Chromatogram - ALD-S1



---AMOUNT UNITS ARE MCG/SPL---

HP 1050 LC S/N 3448A03614

AUTOSAMPLER INJECTION APR 17, 1997 12:42:38

SAMPLE # : ID CODE :

5 31-ALDS1 RR

ALDICARB 50 502 CARBOL 250M 10-10/1-90/16-90/18-10/20 ACN/H2O 1ML/MIN  
40UL CB

ESTD

RT	HEIGHT	AREA	WIDTH	TYPE	UGS	NAME
2.444	18923	286140	.252	BU		
2.991	17260	325168	.314	UP		
4.067	4367	27008	.103	PU		
6.530	6842	113979	.278	PB	.0590	SULFOXIDE
7.640	1495	27472	.306	PB	.0120	SULFONE
10.080	1868	28324	.253	UB		
11.863	2751	17637	.107	UB	.0066	ALDICARB
22.739	8112	249918	.513	I UH		

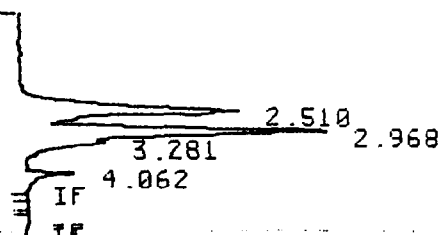
MULTIPLIER = 0.0166675

MLS OF SAMPLE = 0.6667

START PENDING

RUN # 7 APR 17, 1997 13:14:20

START



**Worker Health and Safety  
Laboratory**



**Center for Analytical Chemistry  
3292 Meadowview Road  
Sacramento, California  
916-262-2079**

Air Sample Analysis Report

for

Aldicarb Application



Submitted by:

Sheila Margetich  
Supervisor  
Worker Health and Safety Laboratory

10-3-97



## Table of Contents

---

I. Summary of ARB/CAC Contract	1
Table #1 - ARB Air Sample Log	
II. Analytical Results Record	2-3
Table #2	
III. Summary of WHS Analytical Report	4-8
Table #3 - Application QA Spike Recoveries	7
Table #4 - WHS On-Going QC Spike Recoveries	8
Table #5 - WHS On-Going QC Blank Results	8
IV. Attachments	
A. Copies of ARB Original Chain of Custody Forms	
B. Method SOP	
C. Chromatogram of Seasoned LOD Resin Spike	
D. Standard Curve Chromatograms	
E. WHS-AD-11 SOP Titled "Data Generation and Reporting"	
F. Resin Lab, Field and Trip Spike Chromatograms	
G. Chromatogram of On-Going Resin QC Spike	
H. Chromatogram of On-Going Resin QC Blank Results	
I. Chromatogram of Resin Air Sample	

## **I. Summary of ARB/CAC Contract**

The Worker Health and Safety Laboratory (WHS) of the Center for Analytical Chemistry (CAC) was contracted by the Air Resources Board to perform the analysis of air samples. In partial agreement of that contract, we analyzed 1 set of Aldicarb application samples and accompanying QA samples. Method development and method validation was required for this analyte and its metabolites.

The following table summarizes the samples submitted by ARB for analyses and their analytical completion date. Please see Attachment A1 > A5 for copies of the original chain of custody forms that accompanied these samples.

**TABLE 1. ARB AIR SAMPLE LOG WITH ANALYTICAL COMPLETION DATES**

<b>Date Received</b>	<b>ARB Logbook Numbers (Inclusive)</b>	<b>Total # of air samples</b>	<b>Analysis Completion Date</b>
7-1-97	Aldicarb Application 1-43	35 + QA spikes	7-29-97

TABLE 2.

## ANALYTICAL RESULT RECORD

ARB Aldicarb Application Logbook Number	ARB Field Sample Number	Aldicarb Results * ug/sample	WHS Lab Number
1	WB	ND	WHSC-260
3	SB	No Sample	WHSC-261
5	EB	ND	WHSC-262
7	NB	ND	WHSC-263
9	W1	ND	WHSC-264
10	S1	ND	WHSC-265
11	S1-D	ND	WHSC-266
12	E1	ND	WHSC-267
13	N1	ND	WHSC-268
14	W2	ND	WHSC-269
15	S2	ND	WHSC-270
16	S2-D	ND	WHSC-271
17	E2	ND	WHSC-272
18	N2	ND	WHSC-273
19	W3	ND	WHSC-274
20	S3	ND	WHSC-275
21	S3-D	ND	WHSC-276
22	E3	ND	WHSC-277
23	N3	ND	WHSC-278
24	W4	ND	WHSC-279
25	S4	ND	WHSC-280
26	S4-D	ND	WHSC-281
27	E4	ND	WHSC-282
28	N4	ND	WHSC-283
29	W5	ND	WHSC-284
30	S5	ND	WHSC-285

\* Aldicarb Limit of Detection: 0.05 ug/sample

(2)

ANALYTICAL RESULT RECORD

[illegible]

### III. Summary of WHS Analytical Report

#### 1. SCOPE:

This report covers the WHS analysis of samples labeled Aldicarb Application Log #1-43 (C97-007).

#### 2. SUMMARY OF METHOD:

The analytical method titled "Standard Operating Procedure for the Analysis of Aldicarb and Metabolites on XAD-2 Resin Air Sampling Tubes" as developed and validated by Worker Health and Safety was followed with three exceptions: 1) halfway through the project we had trouble with the Spectra Physics detector and it was replaced with a Hewlett-Packard detector and connected to the HP ChemStation, 2) per ARB, all "None Detected" results were based on the method LOD rather than the method LOQ and 3) to lower the sensitivity, the method was revised to have a final volume of 0.667 mL for the samples. The method LOD was verified by analyzing 5 seasoned spikes at the 0.050 ug/spl level. Please see Attachment B for the Method SOP.

#### WHS Instrumentation

Hewlett Packard 1050 HPLC Liquid Chromatograph

Column: Pickering Laboratories Carbamate Analysis Column, 5  $\mu$ m, C<sub>18</sub>, 4.6 x 250 mm

Post Column System: Pickering PCX 5100

Pump Gradient: ACN/H<sub>2</sub>O - 10%, hold for 2 min., to 90% in 16 min, hold for 2 min, to 10% at 1.0 L/min.

Fluorescence Detector: Spectra Physics Thermo Separation  
excitation at 330 nm  
emission at 464 nm

Retention Times: Aldicarb Sulfoxide	6.50 min.
Aldicarb Sulfone	7.68 min.
Aldicarb	11.87 min.

### 3. ANALYTICAL CALCULATIONS:

A. Calculation of LOD: The Limit of Detection (LOD) for Aldicarb, Aldicarb Sulfoxide and Aldicarb Sulfone was based on at least a 3:1 signal to noise ratio for each of the three peaks. The average peak height for each of these three peaks was 3 mm. At this height, the LOD for Aldicarb was 0.010 µg/spl. The LOD for Aldicarb SO was 0.015 µg/spl. The LOD for Aldicarb SO<sub>2</sub> was 0.025 µg/spl. These LOD calculations were based on a 0.667 mL final volume for each of the ARB samples.

During method development, "seasoned" Aldicarb spikes were prepared and analyzed. These were resin tubes that were spiked with a liquid injection of Aldicarb only and then had air drawn through them for 24 hours at 4L/min. When analyzed, these "seasoned" tubes resulted in both metabolites being present in addition to the Aldicarb parent. Consequently, the LOD for an actual ARB sample was reported as the combined LODs for Aldicarb and its metabolites, i.e. LOD = 0.050 µg/sample.

Calculation of Aldicarb (only) LOD is as follows:

$$\frac{\text{LOD peak height}}{\text{standard peak height}} \times \mu\text{g standard injected} \times \text{uL stand. inj.} \times \frac{\text{sample final volume}}{\mu\text{L sample injected}} = \mu\text{g/sample}$$

#### EXAMPLE:

$$\frac{3 \text{ mm}}{5 \text{ mm}} \times \frac{0.025 \mu\text{g Ald. standard injected}}{\text{mL}} \times 40 \text{ uL stand. inj} \times \frac{0.667 \text{ mL final volume}}{40 \mu\text{L injected}} = 0.010 \mu\text{g/spl}$$

B. Analytical verification of "seasoned" LOD: Five resin tubes (SKC lot # 499) were each spiked by liquid injection with 0.050 µg of Aldicarb (only). The tubes had air drawn through them at 4 L/minute for 24 hours after which they were extracted. Please see Attachment C for the chromatogram of a "seasoned" Aldicarb spike at the 0.050 µg/spl LOD level.

### 3. ANALYTICAL CALCULATIONS: (cont.)

C. Sample result calculations: For reporting of sample results, the total micrograms of Aldicarb and the total micrograms of the two metabolites (corrected for molecular weights) were combined and reported as Aldicarb. Results are reported as total micrograms of Aldicarb per sample. Sample calculation is as follows:

Aldicarb:

$$\frac{3.5\text{mm PH spl}}{3\text{mm PH Ald}} \times \frac{0.025 \mu\text{g Ald.}}{\text{mL}} \times 40 \text{ uL Ald. inj.} \times \frac{1.0 \text{ mL}}{1000 \text{ uL}} \times \frac{0.667 \text{ mL}}{40 \text{ uL spl inj.}} \times \frac{1.0 \text{ mL}}{1000 \text{ uL}} = 0.019 \mu\text{g/sample}$$

Aldicarb SO:

$$\frac{3.5\text{mm PH spl}}{3\text{mm PH Ald SO}} \times \frac{0.025 \mu\text{g SO}}{\text{mL}} \times 40 \text{ uL Ald. inj.} \times \frac{1.0 \text{ mL}}{1000 \text{ uL}} \times \frac{0.667 \text{ mL}}{40 \text{ uL spl inj.}} \times \frac{1.0 \text{ mL}}{1000 \text{ uL}} = 0.019 \mu\text{g/spl SO}$$

$$0.019 \mu\text{g/spl SO} \times \frac{190.25 \text{ M.W. Ald.}}{206.25 \text{ M.W. Ald. SO}} = 0.017 \mu\text{g/sample Aldicarb}$$

Aldicarb SO<sub>2</sub>:

$$\frac{3.5\text{mm PH spl}}{3\text{mm PH Ald SO}_2} \times \frac{0.025 \mu\text{g SO}_2}{\text{mL}} \times 40 \text{ uL Ald. inj.} \times \frac{1.0 \text{ mL}}{1000 \text{ uL}} \times \frac{0.667 \text{ mL}}{40 \text{ uL spl inj.}} \times \frac{1.0 \text{ mL}}{1000 \text{ uL}} = 0.019 \mu\text{g/spl SO}_2$$

$$0.019 \mu\text{g/spl SO}_2 \times \frac{190.25 \text{ M.W. Ald.}}{222.25 \text{ M.W. Ald. SO}_2} = 0.016 \mu\text{g/sample Aldicarb}$$

TOTAL ALDICARB = 0.052  $\mu\text{g/sample}$

Where "PH" = Peak Height

### 4. QUALITY ASSURANCE:

A. Instrument Linearity and Reproducibility: Instrument linearity and reproducibility were evaluated by running 10 replicate 30ul injections of three levels of standards containing Aldicarb, SO and SO<sub>2</sub> over a 13 hour period. The spreadsheets of the ten standard curves are found as attachments to the Method SOP (Attachment B).

B. Standard Curve Linearity and r-value: A three point calibration curve, including 0.05  $\mu\text{g/mL}$ , 0.10  $\mu\text{g/mL}$  and 1.0  $\mu\text{g/mL}$  of Aldicarb, SO and SO<sub>2</sub>, was made to determine the standard curve linearity. Computed r-values for the 10 standard curves were also performed and the plots can be found as attachments in the Method SOP (Attachment B). Before the beginning of sample analysis, a four point calibration curve was established. A standard at the 0.025  $\mu\text{g/mL}$  level was added to cover the lower reportable LOD that was requested by the Study Director. Please see Attachment D1 > D4 for chromatograms of the standard curve levels.

#### 4. QUALITY ASSURANCE: (cont.)

C. Analytical result acceptance criteria: Analytical acceptance criteria based on the linearity and reproducibility of standard curves are detailed in Attachment E, our SOP numbered WHS-AD-11 and titled "Data Generation and Reporting".

##### D. Quality Assurance Spikes:

1. Application QA Spikes: WHS personnel prepared the Quality Assurance spikes for this study since the Center for Analytical Chemistry (CAC) Quality Assurance (QA) personnel were not available at the time. The resin beds of sixteen resin tubes (SKC Lot # 499) were spiked with 70 uL of 10 ng/uL Aldicarb (only) spike solution. This made a total of 700 ng per spike. The standard was secured from CAC Standards Repository. The Standard Solution number was # 72-2922e. After spiking, the tubes were allowed to stand at ambient temperature for about one hour after which the broken ends of the primary sections were capped.

Four tubes were selected at random, extracted and analyzed for spiking level verification. Four additional tubes were retained in the lab in Freezer # 27873 as Lab Spikes. The remaining eight tubes were used by ARB staff as Trip Spikes and Field Spikes. When the tubes were returned to the lab, all 12 tubes were extracted and analyzed concurrently. The following table lists the % recoveries.

**TABLE 3. APPLICATION QA SPIKES - % RECOVERY**

CAC Spike ID	ARB Spike ID	Amount Spiked ng	% Recovery Aldicarb	% Recovery Sulfoxide	% Recovery Sulfone	% Recovery Total Aldicarb
QA-LS-1	---	700	80.57	---	---	80.57
QA-LS-2	---	700	122.8	---	---	122.8
QA-LS-3	---	700	79.43	---	---	79.43
QA-LS-4	---	700	80.14	---	---	80.14
QA-TS-1	TS-1	700	106.8	9.25	---	115.3*
QA-TS-2	TS-2	700	78.71	8.11	---	86.19*
QA-TS-3	TS-3	700	84.57	7.64	---	91.62*
QA-TS-4	TS-4	700	89.71	14.2	---	102.8*
QA-FS-1	WFS1	700	46.86	59.14	9.07	109.2*
QA-FS-2	SFS2	700	50.57	63.14	12.02	119.1*
QA-FS-3	BFS3	700	48.00	51.43	8.63	102.8*
QA-FS-4	NFS4	700	66.00	36.28	5.47	104.1*

\* Total Aldicarb recoveries include the Aldicarb metabolite amounts corrected for molecular weights.

Please see attachment F1 ➤ F3 for resin tube Lab, Trip and Field Spike chromatograms.



## 5. QUALITY CONTROL:

A. Collection efficiencies and storage stability: For collection efficiencies and storage stability data, please refer to Attachment B for the Method SOP as developed by WHS.

B. Resin sample/extract integrity: Once received in the lab, all of the resin samples and spikes were stored in Freezer # 27873. The temperature of this freezer is recorded manually every work day. The average temperature of this freezer during the storage of samples and spikes was -16 ° C. At no time did the temperature vary more than +/- 3 ° C.

In all cases, the resin samples and spikes were analyzed on the same day that they were extracted.

C. On-going Quality Control spikes: The following tables list the WHS Laboratory on-going QC spike recoveries. For each set of 20 samples analyzed, one resin tube was spiked with 50 ng (each) of Aldicarb, SO and SO<sub>2</sub>.

**TABLE 4. WHS LABORATORY ON-GOING QC SPIKES -% RECOVERY AT 50 ng EACH**

Date Analyzed	Lab ID	Sample ID	% Recovery Aldicarb	% Recovery Aldicarb SO	% Recovery Aldicarb SO <sub>2</sub>
7-24-97	Spike 1	Resin Spike	67.60*	92.00	81.20
7-29-97	Spike 2	Resin Spike	93.00	108.8	109.4

\* Spike A has a recovery below acceptable parameters. However, since all of the samples were ND, the samples were not rerun as would normally be the case when low recoveries occur.

Please see Attachment G for a resin spike chromatogram.

D. On-going Quality Control resin blanks: The following table lists the resin blank results that were analyzed as part of the WHS Laboratory on-going QC for this Aldicarb study.

**TABLE 5. WHS LABORATORY ON-GOING QC RESIN BLANK RESULTS**

Date Analyzed	Lab ID	Sample ID	Aldicarb, SO and SO <sub>2</sub>
7-24-97	Blank 1	Resin Blank	ND
7-28-97	Blank 2	Resin Blank	ND

Please see Attachment H for a resin blank chromatogram.

## 6. DISCUSSION:

Please see Attachment I for a chromatogram of an ARB Aldicarb resin sample.

ATTACHMENT A1

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

**ALDICARB APPLICATION  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-007

Date: 7/1/97  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 1-10

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or <u>dry ice</u>
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	7/1/97	1318	KEM	S. Mungin	✓
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
1	WB	
2	WFS1	
3	SB	NO SAMPLE (Broken-KEM)
4	SFS2	
5	EB	
6	EFS3	
7	NB	
8	NFS3	
9	WI	
10	SI	

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DATE 7-1-97 INITIALS SM

*Stored in freezer #2813. Jan 7-1-97*

ATTACHMENT A2

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

ALDICARB APPLICATION  
CHAIN OF CUSTODY

#2

SAMPLE RECORD

Job #: C97-007

Date: 7/1/97  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 11-20

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or dry ice
Sample Collected			KEM		
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	7/1/97	1318	KEM	S. Mayfield	✓
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
11	S1D.	
12	E1.	
13	N1.	
14	W2.	
15	S2.	
16	S2D.	
17	E2.	
18	N2.	
19	W3.	
20	S3.	

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DATE 7-1-97 INITIALS SM

**ATTACHMENT A3**

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

**ALDICARB APPLICATION  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-007

Date: 7/1/97  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 21-30

ACTION			INITIALS <i>KEM</i>		METHOD OF STORAGE freezer, ice or <u>dry ice</u>
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	<i>7/1/97</i>	<i>1318</i>	<i>KEM</i>	<i>S. Magister</i>	<input checked="" type="checkbox"/>
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
<u>21</u>	<u>S30.</u>	
<u>22</u>	<u>E3.</u>	
<u>23</u>	<u>N3.</u>	
<u>24</u>	<u>W4.</u>	
<u>25</u>	<u>S4.</u>	
<u>26</u>	<u>S40.</u>	
<u>27</u>	<u>E4.</u>	
<u>28</u>	<u>N4.</u>	
<u>29</u>	<u>W4.</u>	
<u>30</u>	<u>S5.</u>	

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DATE 7-1-97 INITIALS sm

*Stored in freezer #29813 on 7-1-97*

ATTACHMENT A4

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

**ALDICARB APPLICATION  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-007

Date: 7/1/97  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 31 - 40

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or <u>dry ice</u>
Sample Collected			KEM		
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	5/1/97	1318	KEM	S. Mungin	✓
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
<u>31</u>	<u>SSA.</u>	
<u>32</u>	<u>ES.</u>	
<u>33</u>	<u>NS.</u>	
<u>34</u>	<u>TS1.</u>	
<u>35</u>	<u>TS2.</u>	
<u>36</u>	<u>TS3.</u>	
<u>37</u>	<u>TS4.</u>	
<u>38</u>	<u>B1.</u>	<u>Blank</u>
<u>39</u>	<u>W6.</u>	
<u>40</u>	<u>SB.</u>	

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DATE 7-1-97 INITIALS SM

*Stored in freezer #27873. Sm 1-1-97*

**ATTACHMENT A5**

CALIFORNIA AIR RESOURCES BOARD  
MONITORING & LABORATORY DIVISION  
P.O. Box 2815, Sacramento CA 95812

**ALDICARB APPLICATION  
CHAIN OF CUSTODY**

**SAMPLE RECORD**

Job #: C97-007

Date: 7/1/97  
Sample/Run #: \_\_\_\_\_  
Job name: \_\_\_\_\_  
Log numbers: 41-43

ACTION			INITIALS		METHOD OF STORAGE freezer, ice or <u>dry ice</u>
Sample Collected					
	DATE	TIME	GIVEN BY	TAKEN BY	
Transfer	7/1/97	1318	REM	S. Mungton	✓
Transfer					
Transfer					
Transfer					
Transfer					
Transfer					

LOG #	ID #	DESCRIPTION
<u>41</u>	<u>SGD.</u>	
<u>42</u>	<u>E6.</u>	
<u>43</u>	<u>N6.</u>	

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DATE 7-1-97 INITIALS Sm

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California Department of Food and Agriculture  
Center for Analytical Chemistry  
Worker Health & Safety Laboratory  
3292 Meadowview Road  
Sacramento, CA 95832  
916-262-2079

Original Date: 06-11-97  
Supersedes: New  
Page: 1 of 9

Standard Operating Procedure for the Analysis of  
Aldicarb and Metabolites on XAD-2 Resin Air Sampling Tubes

1. SCOPE:

This is an HPLC post column fluorescence detector method for the determination of Aldicarb, Aldicarb Sulfoxide, and Aldicarb Sulfone on XAD-2 resin air sampling tubes.

2. SUMMARY OF METHOD:

The exposed XAD-2 resin tubes are stored on dry ice or in the freezer until extraction. Samples are equilibrated to room temperature, desorbed with 4 mLs of methanol, and reduced in volume 2:1 prior to injection. The validated injection volume is 100  $\mu$ L, comprised of 30  $\mu$ L of standard or sample and 70  $\mu$ L of water. Depending on system performance, up to 40  $\mu$ L of sample or standard may be injected. The samples are analyzed by HPLC with autosampler, a C18 column, post column system, OPA derivatization, and fluorescence detection.

3. EQUIPMENT AND CONDITIONS:

A. Instrumentation:

Hewlett Packard 1050 Liquid Chromatograph with Autosampler  
Fluorescence Detector: SP Thermo Separation Products Spectra System  
FL2000  
Pickering Post Column System PCX5100  
Column: Pickering Laboratories Carbamate Analysis Column, 5 $\mu$ , C18,  
4.6 x 250 mm  
Data System: Hewlett Packard 3396 Series II Integrator

B. Conditions:

HPLC Pump Gradient: 10% ACN, hold for 2 min, T = 16 min 90% ACN,  
hold for 2 min, T = 20 min 10% ACN, ACN/H<sub>2</sub>O, 1 mL/min,  
Stoptime = 23 min, Posttime = 5 min.

B. Conditions: (cont.)

1050 ALS Injector Program: 1. Draw 30  $\mu\text{L}$  from Sample, Speed 200  $\mu\text{L}/\text{min}$ , Offset 0.0 mm. 2. Draw 70  $\mu\text{L}$  from Sample + 50, Speed 200  $\mu\text{L}/\text{min}$ , Offset 0.0 mm. 3. Mix max  $\mu\text{L}$  needle in seat, Repeat 10 times, Speed 500  $\mu\text{L}/\text{min}$ .

Post Column Parameters: Heated Reactor temperature = 100° C, Column temperature = 42° C, reagent preparation as per Pickering Laboratories PCX5100 User's Manual Version CE 1, January 1997.

Fluorescence detector: excitation at 330 nm, emission at 464 nm.

C. Auxiliary Apparatus:

1. Glass vials and caps, minimum 5 mL capacity
2. Vial Rotator, "Roto-Torque", Heavy Duty Rotator, Cole-Palmer Instrument Company, Chicago, IL
3. Nitrogen Evaporator, Organomation Associates Incorporated, Northborough, MA
4. Miscellaneous laboratory glassware

D. Reagents:

1. Methanol, acetonitrile, and water, HPLC grade
2. 0.05 M NaOH and *o*-phthalaldehyde reagent (OPA) as per Pickering Laboratories PCX5100 5100 User's Manual Version CE.1, January 1997.
3. Analytical standards of Aldicarb, Aldicarb Sulfoxide, and Aldicarb Sulfone in methanol
  - a) Stock standard of 1 mg/mL
  - b) Working standards in the range of 0.05 to 1 ng/ $\mu\text{L}$

4. ANALYSIS OF SAMPLES:

1. Add 4 mLs of methanol to glass extraction vial.
2. Score the primary section end of the sampled XAD-2 tube above the retainer spring and break at the score. Remove the glass wool plug with forceps and place into the extraction vial. Pour the XAD-2 resin into vial. Retain the secondary section of the tube for later analysis if breakthrough is suspected.
3. Rotate or shake vial for 30 minutes.
4. Remove an aliquot of methanol extract and reduce 2:1 under nitrogen. Extract is ready for analysis. If the sample is outside the linear range of the standard curve, concentrate or dilute as necessary.



## 5. QUALITY ASSURANCE

### A. Instrument Linearity and Reproducibility:

Instrument linearity and reproducibility were first evaluated by running 10 replicate 30 uL injections of three levels of standards containing Aldicarb, Aldicarb Sulfoxide (SO), and Aldicarb Sulfone (SO<sub>2</sub>) over a 13 hour period. Please see Attachment 1 ➤ 3 for linearity and reproducibility spreadsheets of Aldicarb, Aldicarb SO and Aldicarb SO<sub>2</sub>. Please see Attachment 4 ➤ 6 for graphs of r-values.

For sample analysis, standard curve linearity and reproducibility are tracked during each analytical run. For a 4 point standard curve ranging from 0.05 ng/μL to 1.0 ng/μL linearity is acceptable if  $r \geq 0.995$  and if instrument response for a given level is within  $\pm 20\%$  of its calculated value.

Instrument reproducibility is acceptable if responses for a given standard curve fall within  $\pm 20\%$  of the preceding curve.

### B. Limits of Detection and Quantitation:

The Limit of Detection (LOD) for Aldicarb was based on at least a 3:1 signal to noise ratio\*\* and an average peak height of 7 mm for the lowest standard of 0.05 ng/μL. Calculation is as follows:

$$\frac{\text{LOD peak height}}{\text{standard peak height}} \times \text{ng standard injected} \times \frac{\text{final volume}}{\mu\text{L sample injected}} = \mu\text{g/sample}$$

For example:

$$\frac{3 \text{ mm}}{7 \text{ mm}} \times 1.5 \text{ ng standard injected} \times \frac{2 \text{ mL final volume}}{30 \mu\text{L injected}} = 0.043 \mu\text{g/sample}$$

The Limit of Quantitation (LOQ), \*\* based on ten times the signal to noise to ratio, or 3.33 x the LOD, is:

$$3.33 \times .043 = 0.143 \text{ or } 0.140 \mu\text{g/sample}$$

As per the above calculation, the LODs for Aldicarb Sulfoxide and Sulfone were based on average peak heights of 5 mm and 6 mm, respectively. This results in an LOD of 0.060 μg/sample and an LOQ of 0.200 μg/sample for the Sulfoxide and an LOD of 0.050 μg/sample and an LOQ of 0.165 μg/sample for the Sulfone.

## B. Limits of Detection and Quantitation: (cont.)

For actual sample analysis, the total micrograms of the three analytes may be reported.

Results are reported as micrograms per sample. Calculation is:

$$\mu\text{g/sample} = \frac{(\text{sample peak height})(\mu\text{L of std injected})(\text{sample final volume, in mLs})}{(\text{std peak height})(\mu\text{L of sample injected})}$$

\*\* See Standard Operating Procedure WHS-QA-1, revision #1.

## C. Extraction and Collection Efficiencies:

The following table lists the extraction efficiencies of Aldicarb (only) at the method LOQ, 2 x LOQ and 5 x LOQ from unseasoned XAD tubes.

TABLE 1. EXTRACTION EFFICIENCIES OF UNSEASONED XAD TUBES

Level	Spike ID	Amount spiked ng	Amount Recovered ng	% Recovery
1 x LOQ	Rep #1	140.0	121.7	86.93
1 x LOQ	Rep #2	140.0	117.0	83.57
1 x LOQ	Rep #3	140.0	111.0	79.29
1 x LOQ	Rep #4	140.0	105.6	75.43
1 x LOQ	Rep #5	140.0	108.0	77.14
				AVG.: 80.47
2 x LOQ	Rep #1	280.0	235.1	83.96
2 x LOQ	Rep #2	280.0	262.8	93.86
2 x LOQ	Rep #3	280.0	222.0	79.29
2 x LOQ	Rep #4	280.0	Lab Accident	Lab Accident
2 x LOQ	Rep #5	280.0	235.2	84.00
				AVG.: 85.28
5 x LOQ	Rep #1	700.0	638.3	91.19
5 x LOQ	Rep #2	700.0	582.6	83.23
5 x LOQ	Rep #3	700.0	637.3	91.04
5 x LOQ	Rep #4	700.0	684.0	97.71
5 x LOQ	Rep #5	700.0	615.0	87.86
				AVG.: 90.21

C. Extraction and Collection Efficiencies: (cont.)

The following table lists the collection and extraction efficiencies of Aldicarb (only) at the method LOQ, 2 x LOQ and 5 x LOQ from seasoned XAD tubes. The tubes were spiked and then subjected to an air flow of 4L/min for 24 hours at an ambient temperature of about 85 ° F before the primary section was extracted and analyzed.

**TABLE 2. COLLECTION AND EXTRACTION EFFICIENCIES OF SEASONED XAD TUBES**

Level	Spike ID	Amount spiked ng	Amount Recovered ng	% Recovery
1 x LOQ	Rep #1	140.0	141.7	101.21
1 x LOQ	Rep #2	140.0	125.1	89.36
1 x LOQ	Rep #3	140.0	144.2	103.00
1 x LOQ	Rep #4	140.0	169.8	121.29
1 x LOQ	Rep #5	140.0	126.3	90.21
				AVG.: 101.0
2 x LOQ	Rep #1	280.0	331.1	118.25
2 x LOQ	Rep #2	280.0	311.3	111.18
2 x LOQ	Rep #3	280.0	290.3	103.68
2 x LOQ	Rep #4	280.0	380.2	135.79*
2 x LOQ	Rep #5	280.0	332.7	118.82
				AVG.: 113.0
5 x LOQ	Rep #1	700.0	697.7	99.67
5 x LOQ	Rep #2	700.0	Lab Accident	Lab Accident
5 x LOQ	Rep #3	700.0	735.3	105.04
5 x LOQ	Rep #4	700.0	683.2	97.60
5 x LOQ	Rep #5	700.0	567.9	81.13
				AVG.: 95.86

\* This data point was not used to compute the average. It is believed to have been the result of a calibration problem.

D. Storage Stability:

Two sets of 15 resin tube storage stability spikes were done. One set was spiked with 50 ng/spl of Aldicarb. The other set was spiked with 100 ng/spl of Sulfoxide and Sulfone (each). All tubes were spiked at the same time. They were unseasoned per the study director. One set of tubes was extracted and analyzed immediately (Time 0), the remainder of the tubes were placed in freezer #27873.

Spikes were analyzed in triplicate at Time 0 = extracted immediate spiking, at Time 1 = day 8, at Time 2 = day 15, at Time 3 = day 22, and at Time 4 = day 30. Time 4 extracts were rerun on day 56 due to unacceptable metabolite integration on day 30. The following tables (3,4,5) list the storage spike recoveries.

TABLE 3. STORAGE STABILITY SPIKES - 50 ng/spl of Aldicarb

Time	Spike ID	ng Spiked	ng Recovered	% Recovery	Mean	S. D.	% CV
Day 0	SS Spike #1	50.0	44.8	89.60			
	SS Spike #2	50.0	41.7	83.40			
	SS Spike #3	50.0	43.6	87.20	86.73	3.13	3.61
Day 8	SS Spike #1	50.0	52.7	105.4			
	SS Spike #2	50.0	48.0	96.00			
	SS Spike #3	50.0	47.5	95.00	98.80	5.74	5.81
Day 15	SS Spike #1	50.0	44.5	89.00			
	SS Spike #2	50.0	51.1	102.2			
	SS Spike #3	50.0	43.3	86.60	92.60	8.40	9.07
Day 22	SS Spike #1	50.0	37.4 *	74.80			
	SS Spike #2	50.0	36.7	73.40			
	SS Spike #3	50.0	54.4 **	108.8	85.67	20.05	23.40
Day 56	SS Spike #1	50.0	53.1	106.2			
	SS Spike #2	50.0	46.5	93.00			
	SS Spike #3	50.0	42.4	84.80	94.67	10.80	11.41

\* This spike had 9.9 ng/spl of Aldicarb Sulfoxide. Total Recovery = 94.60%

\*\* This spike had 6.3 ng/spl of Aldicarb Sulfoxide. Total Recovery = 121.40 %

Original Date: 06-11-97

Supersedes: New

Page: 7 of 9

D. Storage Stability: (cont.)

TABLE 4. STORAGE STABILITY SPIKES - 100 ng/spl of Aldicarb SO

Time	Spike ID	ng Spiked	ng Recovered	% Recovery	Mean	S. D.	% CV
Day 0	SS Spike #1	100.0	110.7	110.7			
	SS Spike #2	100.0	102.9	102.9			
	SS Spike #3	100.0	87.6	87.60	100.4	11.75	11.70
Day 8	SS Spike #1	100.0	100.1	100.1			
	SS Spike #2	100.0	96.1	96.10			
	SS Spike #3	100.0	132.1	132.1	109.43	19.73	18.03
Day 15	SS Spike #1	100.0	104.9	104.9			
	SS Spike #2	100.0	103.5	103.5			
	SS Spike #3	100.0	88.7	88.70	99.03	8.98	9.07
Day 22	SS Spike #1	100.0	103.4	103.4			
	SS Spike #2	100.0	123.8	123.8			
	SS Spike #3	100.0	88.9	88.90	105.37	17.53	16.64
Day 56	SS Spike #1	100.0	94.9	94.90			
	SS Spike #2	100.0	95.0	95.0			
	SS Spike #3	100.0	113.3	113.3	101.07	10.59	10.48

D. Storage Stability: (cont.)

TABLE 5. STORAGE STABILITY SPIKES - 100 ng/spl of Aldicarb SO<sub>2</sub>

Time	Spike ID	ng Spiked	ng Recovered	% Recovery	Mean	S. D.	% CV
Day 0	SS Spike #1	100.0	109.3	109.3			
	SS Spike #2	100.0	102.6	102.6			
	SS Spike #3	100.0	97.1	97.10	103.00	6.11	5.93
Day 8	SS Spike #1	100.0	106.2	106.2			
	SS Spike #2	100.0	103.0	103.0			
	SS Spike #3	100.0	132.5	132.5	113.90	16.19	14.21
Day 15	SS Spike #1	100.0	100.0	100.0			
	SS Spike #2	100.0	100.4	100.4			
	SS Spike #3	100.0	91.8	91.80	97.40	4.85	4.98
Day 22	SS Spike #1	100.0	105.5	105.5			
	SS Spike #2	100.0	116.7	116.7			
	SS Spike #3	100.0	109.2	109.2	110.47	5.71	5.17
Day 56	SS Spike #1	100.0	94.6	94.60			
	SS Spike #2	100.0	94.5	94.50			
	SS Spike #3	100.0	105.0	105.0	98.03	6.03	6.15

6. DISCUSSION:

Seasoned spiking shows that Aldicarb metabolizes to its Sulfoxide and Sulfone under actual sample conditions. Therefore, the three analytes are included in all QC measures.

The validation of this method included spiking at the LOQ, 2 x LOQ, and 5 x LOQ. Breakthrough was not observed at any level.

The Hewlett Packard 1050 Autosampler Injector Program was used to combine and mix 30 µL of standard or sample with 70 µL of water before injection. This way, the solvent composition of the sample more closely approximates the mobile phase, allowing larger injection volumes and greater sensitivity. Although no mixing takes place when the combined injection volumes equal the capacity of the sample loop, peak shape is substantially improved.

Original Date: 06-11-97  
Supersedes: New  
Page: 9 of 9

Written By:

*Carolinda Benson*

Carolinda Benson, Agricultural Chemist  
Worker Health & Safety Laboratory

Approved By:

*Sheila Margetich*

Sheila Margetich, Supervisor  
Worker Health & Safety Laboratory

Reviewed By:

*Joe Taluk For Terry Jackson*

Terry Jackson, QA Officer  
Center for Analytical Chemistry

Project: ARB Aldicarb and Metabolites on XAD-2 Resin Air Sampling Tubes  
 Chromatogram I.D.: ARB-6b 3-21-22-97 WHS-QA-2 #3  
 Instrument: 1050LC/PCX 5100 spl30/70H2O LC/Mth1 C18 25cm ex330em464 aldica40 dat

# ATTACHMENT I

## Aldicarb Sulfone

	Conc	rt	Area	Int	Calx10-5*	Lin**	Repro***	
Curve 1	0.05	7.669	58766	vb	2.552			
r = 0.99997	0.1	7.661	102599	pb	2.924	87.29%		
	1	7.640	1049174	bb	2.859	102.26%		
Curve 2	0.05	7.645	63130	pb	2.376		107.43%	
r = 0.99996	0.1	7.635	106073	pb	2.828	84.01%	103.39%	
	1	7.635	1060317	pb	2.829	99.96%	101.06%	
Curve 3	0.05	7.642	55070	vb	2.722		87.23%	
r = 0.99998	0.1	7.640	115010	pb	2.608	104.42%	108.43%	
	1	7.645	1063625	bb	2.821	92.48%	100.31%	
Curve 4	0.05	7.640	68200	pb	2.199		123.84%	
r = 0.99992	0.1	7.640	103390	vb	2.902	75.80%	89.90%	
	1	7.638	1056363	bb	2.840	102.17%	99.32%	
Curve 5	0.05	7.635	56417	vb	2.659		82.72%	
r = 1.0000	0.1	7.630	111472	vb	2.691	98.79%	107.82%	
	1	7.641	1079407	pb	2.779	96.83%	102.18%	
Curve 6	0.05	7.639	54675	pb	2.743		96.91%	
r = 0.99999	0.1	7.629	114632	pb	2.617	104.83%	102.83%	
	1	7.641	1078054	pb	2.783	94.04%	99.87%	
Curve 7	0.05	7.638	50547	pb	2.968		92.45%	
r = 0.99997	0.1	7.625	113937	pb	2.633	112.70%	99.39%	
	1	7.647	1087786	pb	2.758	95.47%	100.90%	
Curve 8	0.05	7.649	59330	vb	1.528		117.38%	
r = 1.0000	0.1	7.634	112712	pb	2.662	94.99%	98.92%	
	1	7.640	1081926	bb	2.773	95.99%	99.46%	
Curve 9	0.05	7.643	51669	pb	2.903		87.09%	
r = 0.99985	0.1	7.650	126309	vb	2.375	122.23%	112.06%	
	1	7.643	1093214	pb	2.744	86.55%	101.04%	
Curve 10	0.05	7.639	64566	vb	2.323		124.96%	Repro with Curve 1
r = 1.0000	0.1	7.642	118106	vb	2.540	91.46%	93.51%	109.87%
	1	7.637	1096886	pb	2.735	92.87%	100.34%	114.44%
								115.11%
								105.95%
								104.55%
								101.62%

ARB WB10

\*Calibration Factor: Calculated by HP 3396 Series II Integrator. Is equal to ngs of standard divided by peak area.

\*\*Linearity: Calculated by Quattro Pro. Sample calculation : (peak area for .1ng/ul standard divided by 2 x peak area for .05ng/ul standard) x 100

\*\*\* Reproducibility: Calculated by Quattro Pro. Sample calculation : (peak area for .05ng/ul concentration of 2nd standard curve divided by peak area for .05ng/ul concentration for 1st standard curve) x 100



Project: ARB Aldicarb and Metabolites on XAD-2 Resin Air Sampling Tubes  
 Chromatogram I.D.: ARB-6b 3-21,22-97 WHS-QA-2 #3  
 Instrument: 1050LC/PCX 5100 spl30/70H2O LC/Mth1 C18 25cm ex330em464 aldica40 dat

# ATTACHMENT 2

## Aldicarb Sulfoxide

	Conc	rt	Area	Int	Calx10-5*	Lin**	Repro***	
Curve 1	0.05	6.562	58368	vb	2.570			
r = 0.99995	0.1	6.553	94853	pb	3.163	81.25%		
	1	6.516	935982	pb	3.205	98.68%		
Curve 2	0.05	6.523	46782	pb	3.206		80.15%	
r = 0.99999	0.1	6.505	99710	pb	3.009	106.57%	105.12%	
	1	6.502	965815	pb	3.106	96.86%	103.19%	
Curve 3	0.05	6.505	55070	vb	2.724		117.72%	
r = 0.99998	0.1	6.504	96408	pb	3.112	87.53%	96.69%	
	1	6.508	958915	pb	3.129	99.46%	99.29%	
Curve 4	0.05	6.515	34034	pb	4.407		61.80%	
r = 0.99983	0.1	6.5069	101524	pb	2.995	149.15%	105.31%	
	1	6.502	964603	pb	3.110	95.01%	100.59%	
Curve 5	0.05	6.501	45917	pb	3.267		134.92%	
r = 1.0000	0.1	6.495	98090	pb	3.058	106.81%	96.62%	
	1	6.514	988366	bb	3.035	100.76%	102.46%	
Curve 6	0.05	6.503	51072	pb	2.937		111.23%	
r = 1.0000	0.1	6.493	100992	pb	2.971	98.87%	102.96%	
	1	6.506	960213	pb	3.124	95.08%	97.15%	
Curve 7	0.05	6.527	61743	pb	2.492		120.89%	
r = 0.99994	0.1	6.493	98534	pb	3.045	79.79%	97.57%	
	1	6.505	958507	pb	3.130	97.28%	99.82%	
Curve 8	0.05	6.522	57167	pb	2.624		92.59%	
r = 0.99992	0.1	6.486	93604	pb	3.205	81.87%	95.00%	
	1	6.512	992581	pb	3.022	106.04%	103.55%	
Curve 9	0.05	6.474	57140	vb	2.625		99.95%	
r = 1.0000	0.1	6.513	104939	pb	2.859	91.83%	112.11%	
	1	6.513	984122	pb	3.048	93.78%	99.15%	
Curve 10	0.05	6.522	71536	vb	2.097		125.19%	Repro with Curve 1
r = 0.99987	0.1	6.516	103611	pb	2.8985	72.42%	98.73%	Repro with Curve 5
	1	6.501	994820	pb	3.016	96.01%	101.09%	122.56%
								109.23%
								105.63%
								106.29%
								100.65%

\*Calibration Factor: Calculated by HP 3396 Series II Integrator. Is equal to ngs of standard divided by peak area.

\*\*Linearity: Calculated by Quattro Pro. Sample calculation : (peak area for .1ng/ul standard divided by 2 x peak area for .05ng/ul standard) x 100

\*\*\* Reproducibility: Calculated by Quattro Pro. Sample calculation : (peak area for .05ng/ul concentration of 2nd standard curve divided by peak area for .05ng/ul concentration for 1st standard curve) x 100

Project: ARB Aldicarb and Metabolites on XAD-2 Resin Air Sampling Tubes  
 Chromatogram I.D.: ARB-6b 3-21.22-97 WHS-QA-2 #3  
 Instrument: 1050LC/PCX 5100 spl30/70H2O LC/Mth1 C18 25cm ex330em464 aldica40.dat

# ATTACHMENT 3

Aldicarb							
	Conc	rt	Area	Int	Cal x -5*	Lin**	Repro***
Curve 1	0.05	11.841	64765	pb	2.316		
r = 0.99999	0.1	11.846	130860	pb	2.293	101.03%	
	1	11.818	1235078	pb	2.429	94.38%	
Curve 2	0.05	11.836	58561	pb	2.561		90.42%
r = 0.99998	0.1	11.829	130259	pb	2.303	111.22%	99.54%
	1	11.824	1269031	pb	2.364	97.42%	102.75%
Curve 3	0.05	11.831	71750	pb	2.091		122.52%
r = 0.99995	0.1	11.824	121771	pb	2.464	84.86%	93.48%
	1	11.827	1274999	pb	2.353	104.70%	100.47%
Curve 4	0.05	11.828	76053	pb	1.972		106.00%
r = 0.99999	0.1	11.825	131398	pb	2.283	86.39%	107.91%
	1	11.824	1252398	pb	2.395	95.31%	98.23%
Curve 5	0.05	11.831	74189	pb	2.022		97.55%
r = 0.99999	0.1	11.816	132701	pb	2.261	89.43%	100.99%
	1	11.823	1308502	pb	2.293	98.61%	104.48%
Curve 6	0.05	11.825	75276	vb	1.993		101.47%
r = 0.99998	0.1	11.829	130805	vb	2.293	86.88%	98.57%
	1	11.836	1291564	pb	2.323	98.74%	98.71%
Curve 7	0.05	11.837	79700	pb	1.882		105.88%
r = 1.0000	0.1	11.829	141210	vb	2.124	88.59%	107.95%
	1	11.841	1279144	pb	2.345	90.58%	99.04%
Curve 8	0.05	11.840	62213	pb	2.411		78.06%
r = .99994	0.1	11.822	140277	pb	2.139	112.74%	99.34%
	1	11.821	1264511	pb	2.372	90.14%	98.86%
Curve 9	0.05	11.832	78348	vb	1.915		125.94%
r = 0.99999	0.1	11.843	137133	pb	2.188	87.52%	97.76%
	1	11.838	1297430	pb	2.312	94.61%	102.60%
Curve 10	0.05	11.826	73077	vb	2.053		93.27%
r = 0.99992	0.1	11.838	154440	pb	1.943	105.67%	112.62%
	1	11.833	1301573	pb	2.305	84.28%	100.32%
							Repro with Curve 1
							Repro with Curve 5
							112.83%
							98.50%
							118.02%
							116.38%
							105.38%
							99.47%

ARB.WB18

\*Calibration Factor: Calculated by HP 3396 Series II Integrator. Is equal to ngs of standard divided by peak area.

\*\*Linearity: Calculated by Quattro Pro. Sample calculation : (peak area for .1ng/ul standard divided by 2 x peak area for .05ng/ul standard) x 100

\*\*\* Reproducibility: Calculated by Quattro Pro. Sample calculation : (peak area for .05ng/ul concentration of 2nd standard curve divided by peak area for .05ng/ul concentration for 1st standard curve) x 100

## WHS LABORATORY METHOD VALIDATION FORM

PROJECT: ARBSTUDY DIRECTOR: Kevin MongarP.A.I.: Sheila MargetichSOP: WHS-QA-2, rev. #5, #3LAB BOOK: S96173

## Instrument Linearity Check

Instrument: Hewlett Packard 1050 HPLC S/N 3448A03614 with Pickering PCX 5100  
Post Column System

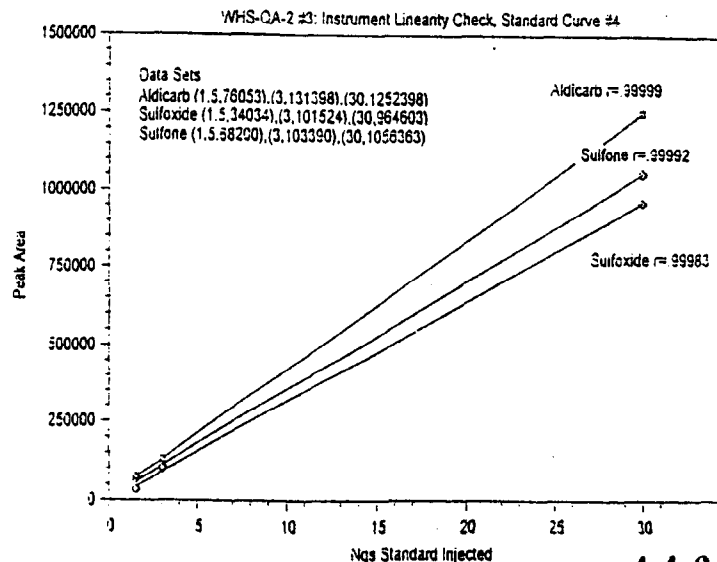
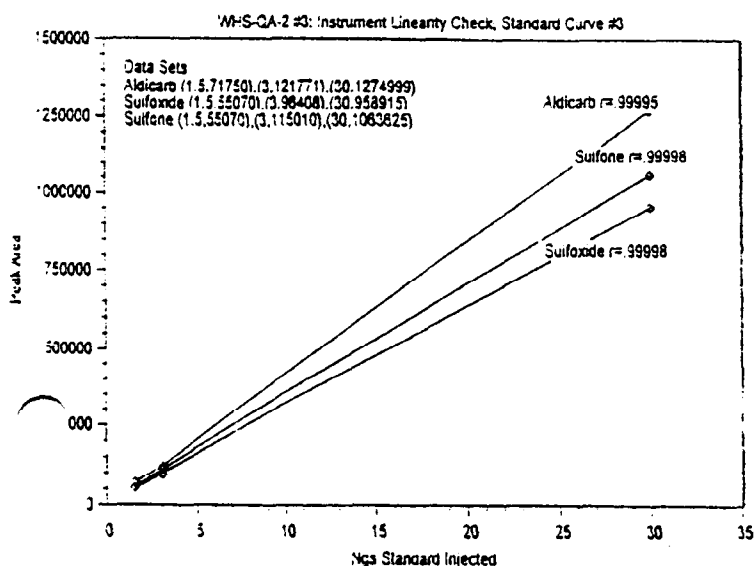
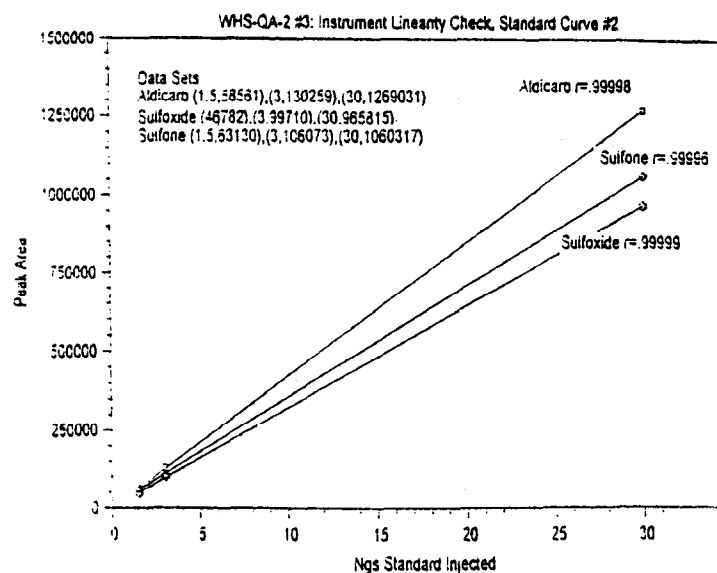
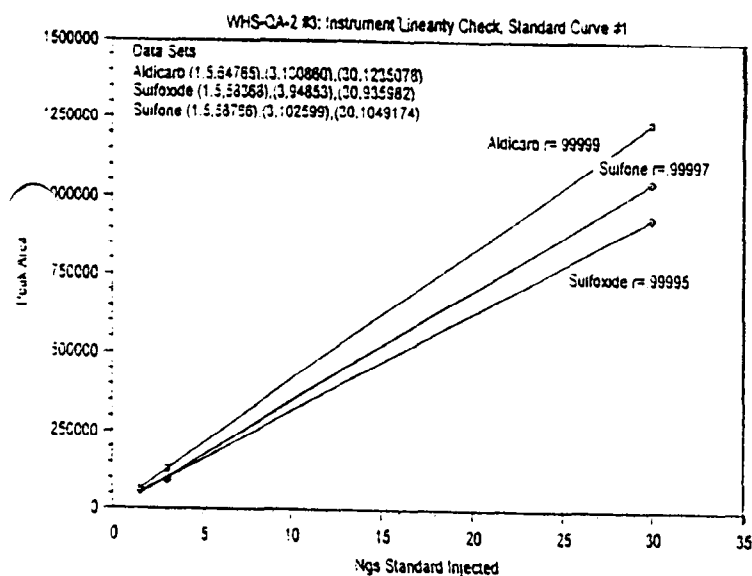
Detector: Spectra Physics Fluorescence, em 330nm/ex 464nm

Analyte(s): Aldicarb, Aldicarb Sulfoxide, Aldicarb Sulfone standards in methanol

Chromatogram ID: ARB-6b

Date of Linearity Check: 3-21, 22-97

$$r = \frac{n \sum xy - \sum x \sum y}{\sqrt{\left[ n \sum x^2 - (\sum x)^2 \right] \left[ n \sum y^2 - (\sum y)^2 \right]}}$$



## WHS LABORATORY METHOD VALIDATION FORM

PROJECT: ARB  
 STUDY DIRECTOR: Kevin Mongar  
 P.A.I.: Sheila Margetich

SOP: WHS-QA-2, rev. #5, #3  
 LAB BOOK: S96173

## Instrument Linearity Check

Instrument: Hewlett Packard 1050 HPLC S/N 3448A03614 with Pickering PCX 5100  
 Post Column System

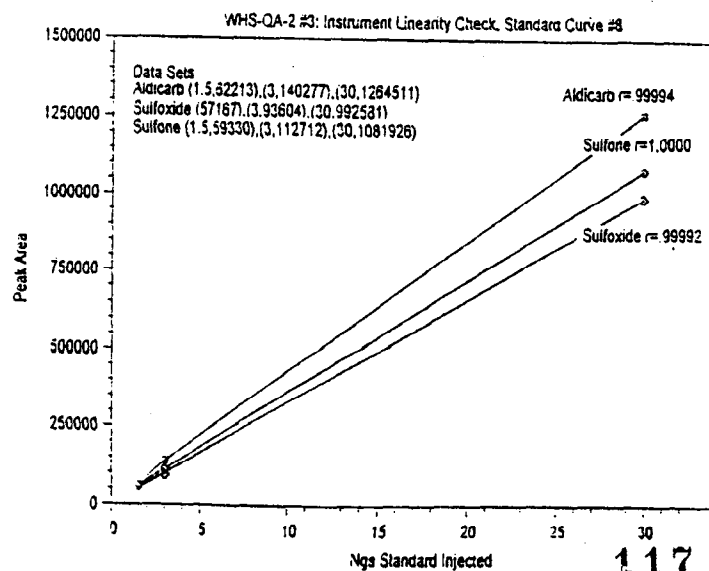
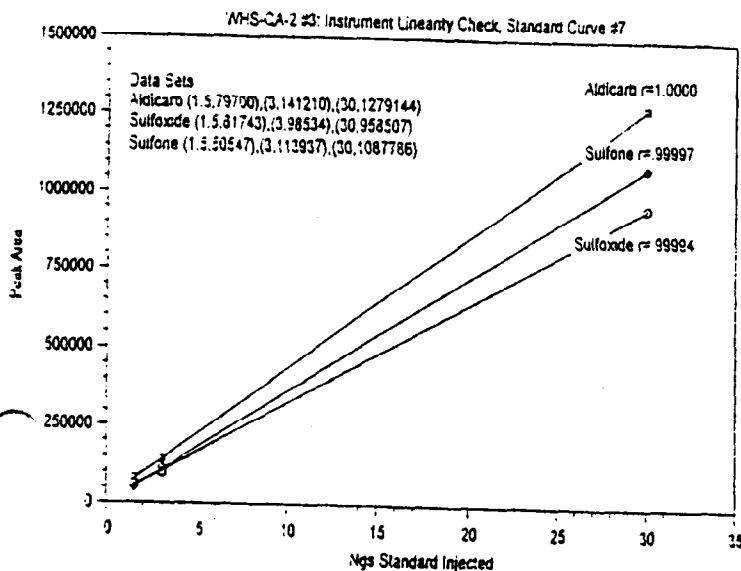
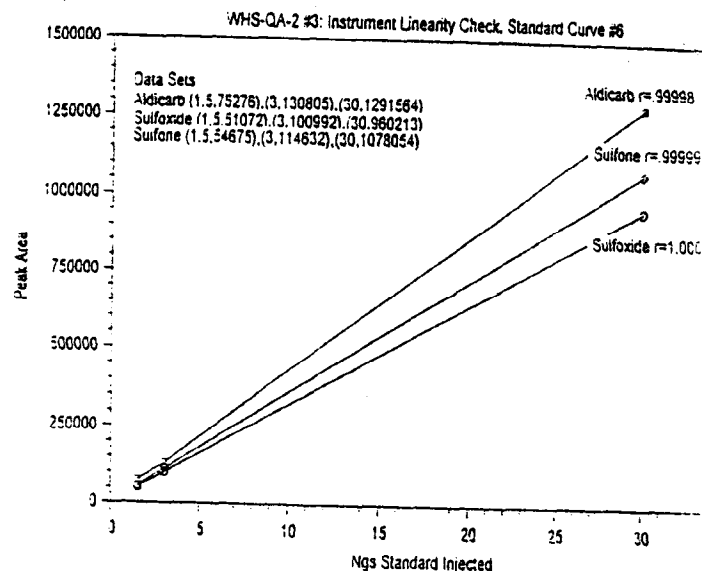
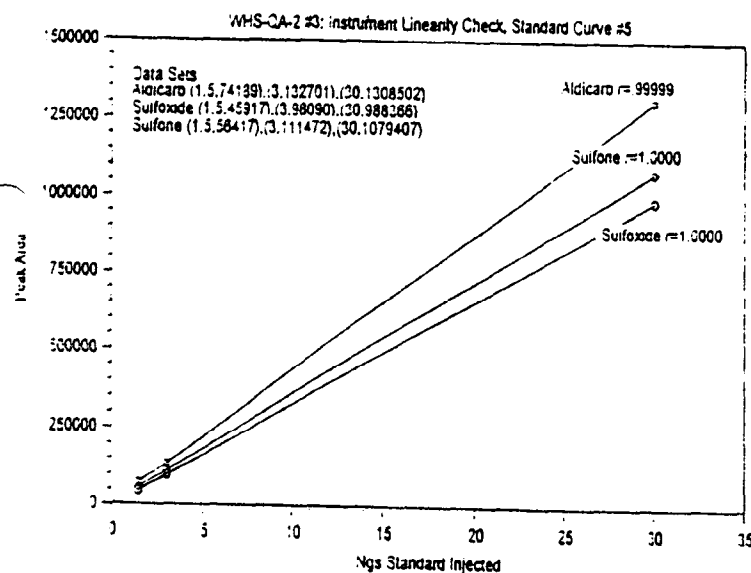
Detector: Spectra Physics Fluorescence, em 330nm/ex 464nm

Analyte(s): Aldicarb, Aldicarb Sulfoxide, Aldicarb Sulfone standards in methanol

Chromatogram ID: ARB-6b

Date of Linearity Check: 3-21, 22-97

$$r = \frac{n \sum xy - \sum x \sum y}{\sqrt{\left\{ n \sum x^2 - (\sum x)^2 \right\} \left\{ n \sum y^2 - (\sum y)^2 \right\}}}$$



## WHS LABORATORY METHOD VALIDATION FORM

PROJECT: ARBSTUDY DIRECTOR: Kevin MongarP.A.I.: Sheila MargetichSOP: WHS-QA-2, rev. #5, #3LAB BOOK: S96173

## Instrument Linearity Check

Instrument: Hewlett Packard 1050 HPLC S/N 3448A03614 with Pickering PCX 5100  
Post Column System

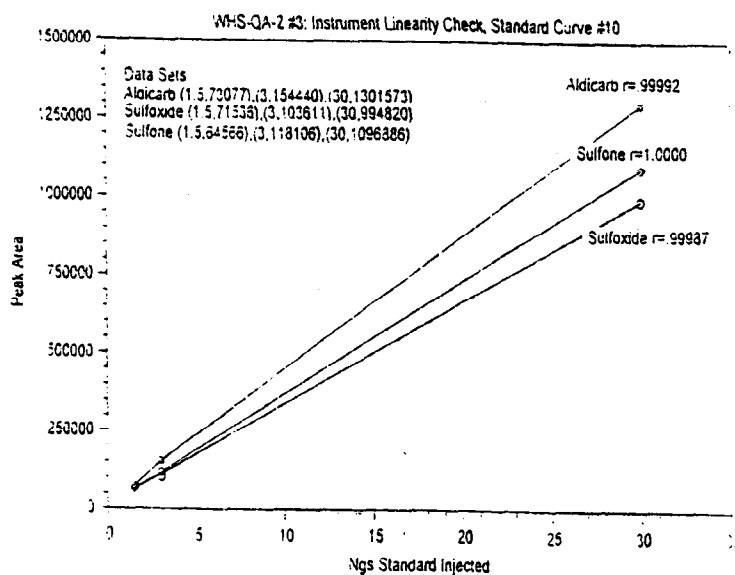
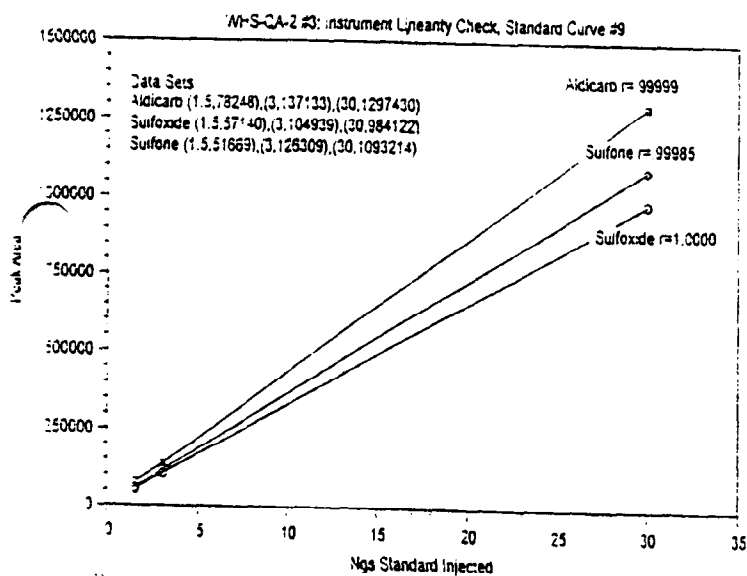
Detector: Spectra Physics Fluorescence, em 330nm/ex 464nm

Analyte(s): Aldicarb, Aldicarb Sulfoxide, Aldicarb Sulfone standards in methanol

Chromatogram ID: ARB-6b

Date of Linearity Check: 3-21, 22-97

$$r = \frac{n \sum xy - \sum x \sum y}{\sqrt{\left[ n \sum x^2 - (\sum x)^2 \right] \left[ n \sum y^2 - (\sum y)^2 \right]}}$$



Data File

ATTACHMENT C  
Seasoned LOD Resin Spike - 50 ng/spl

Name:

Injection Date : 6/2/97 2:50:28 PM

Seq. Line : 6

Sample Name : 50-2

Vial : 106

Acq. Operator : cb

Inj : 1

Inj Volume : Inj prog

Acq. Method : C:\HPCHEM\1\METHODS\CARB.M

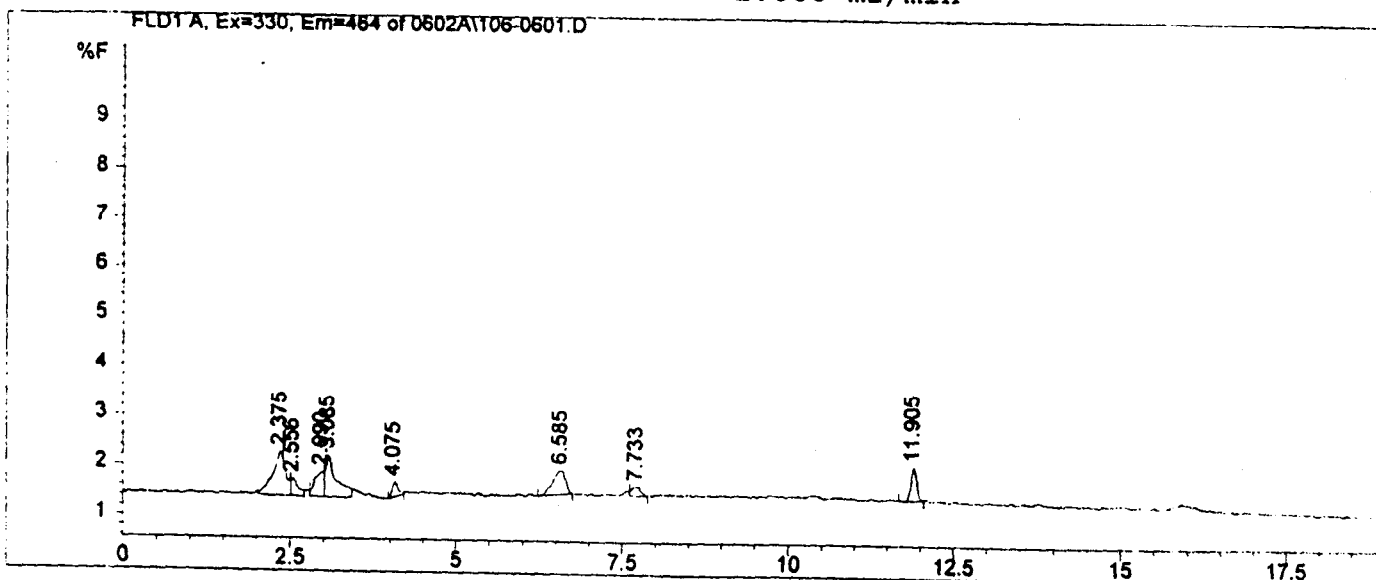
Last changed : 6/2/97 2:10:08 PM by cb

Analysis Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 6/3/97 10:51:10 AM by cb

(modified after loading)

Instrument Conditions: At Start At Stop  
Temperature: 24.3 24.4 °C  
Pressure: 165.2 165.4 bar  
Flow: 1.000 1.000 ml/min



External Standard Report

Sorted by Retention Time

Calib. Data Modified : Tuesday, June 03, 1997 10:44:19 AM

Multiplier : 1.667000e-2

Dilution : 1.000000

Uncalibrated Peaks : compound name not specified

Signal 1: FLD1 A, Ex=330, Em=464

Injection Date : 6/2/97 2:50:28 PM

Seq. Line : 6

Sample Name : 50-2

Vial : 106

Acq. Operator : cb

Inj : 1

Inj Volume : Inj prog

Acq. Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 6/2/97 2:10:08 PM by cb

Analysis Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 6/3/97 10:51:10 AM by cb

(modified after loading)

RT [min]	Sig	Type	Area [%F*s]	Amt/Area	Amount [ug/spl]	Grp	Name
2.375	1	VV	12.04306	0.00000	0.00000		?
2.556	1	VV	2.56568	0.00000	0.00000		?
2.990	1	VV	4.56051	0.00000	0.00000		?
3.085	1	VV	9.73752	0.00000	0.00000		?
4.075	1	PV	1.90652	0.00000	0.00000		?
6.585	1	PV	6.61181	2.97628e-1	3.28043e-2		sulfoxide
7.733	1	VV	1.79836	3.02531e-1	9.06950e-3		sulfone $1.92 \times \text{Inj std} \times \frac{u}{4}$
11.905	1	PV	4.14061	2.18828e-1	1.51043e-2		aldicarb $5.02$
22.581	1	VV	2.62287	0.00000	0.00000		?
22.667	1	VV	1.00735	0.00000	0.00000		?

Totals :

5.69781e-2

$= .00548 \text{ ug/spl}$   
 CB  $6-30$   
 6-3-97

\*\*\* End of Report \*\*\*

# ATTACHMENT D-1

Standard Curve Chromatogram - 0.025 ng/ul

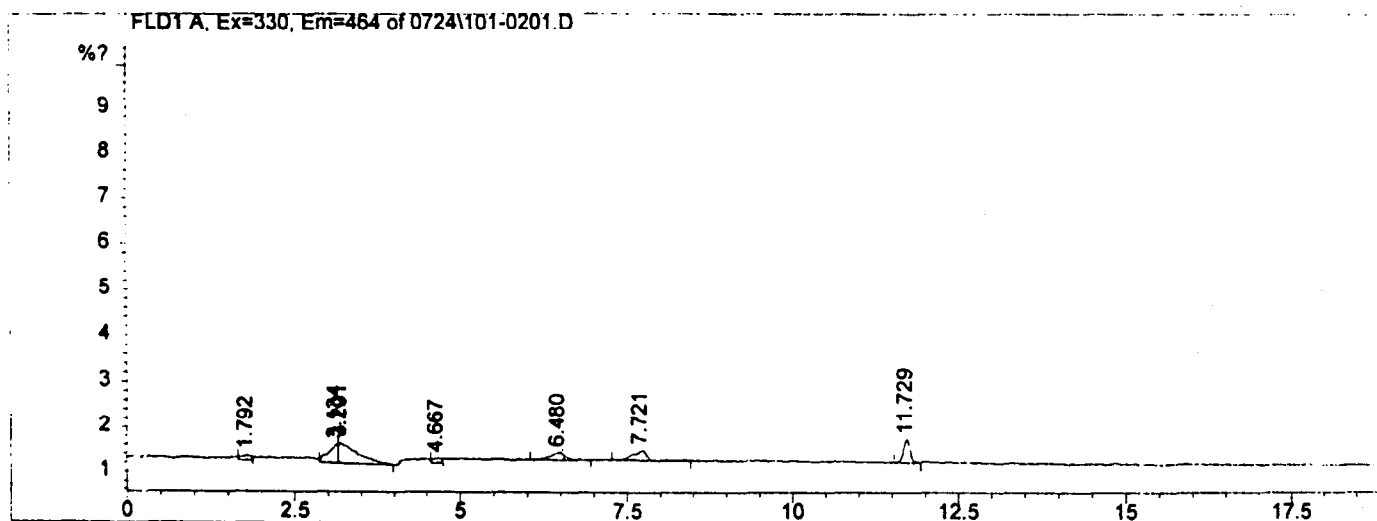
.025 ng

Injection Date : 7/24/97 4:02:09 PM Seq. Line : 2  
 Sample Name : .025 ng/ul Vial : 101  
 Acq. Operator : sf Inj : 1  
 Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S  
 Acq. Method : C:\HPCHEM\1\METHODS\CARB.M  
 Last changed : 7/24/97 3:58:38 PM by sf  
 (modified after loading)  
 Analysis Method : C:\HPCHEM\1\METHODS\CARB.M  
 Last changed : 7/24/97 4:25:26 PM by sf  
 (modified after loading)

carbamate method

Instrument Conditions: At Start At Stop  
 Temperature: 27.6 27.7 °C  
 Pressure: 154.0 155.6 bar  
 Flow: 1.000 1.000 ml/min



## Calibration Table (after recalibration)

Calib. Data Modified : Thursday, July 24, 1997 4:25:26 PM

Level 1 calibrated: Replace Response Factors,  
 No Update of Retention Times

Signal 1 : FLD1 A, Ex=330, Em=464



Injection Date : 7/24/97 4:02:09 PM

Seq. Line : 2

Sample Name : .025 ng/ul

Vial : 101

Acq. Operator : sf

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Acq. Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/24/97 3:58:38 PM by sf  
(modified after loading)

Analysis Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/24/97 4:25:26 PM by sf  
(modified after loading)

carbamate method

RT [min]	Sig	Lvl	Amount [ug/spl]	Area	Amt/Area	Ref Grp	Name
6.558	1	1	1.00000	2.67483	3.73855e-1		sulfoxide
		2	2.00000	5.81434	3.43977e-1		
		3	4.00000	10.31539	3.87770e-1		
		4	40.00000	105.60493	3.78770e-1		
7.755	1	1	1.00000	3.10021	3.22559e-1		sulfone
		2	2.00000	6.24733	3.20137e-1		
		3	4.00000	11.61346	3.44428e-1		
		4	40.00000	116.78109	3.42521e-1		
11.757	1	1	1.00000	3.70002	2.70269e-1		aldicarb
		2	2.00000	7.25617	2.75628e-1		
		3	4.00000	13.94594	2.86822e-1		
		4	40.00000	139.17429	2.87409e-1		

Rel. Reference Window : 8.000 %

Abs. Reference Window : 0.000 min

Rel. Non-ref. Window : 8.000 %

Abs. Non-ref. Window : 0.000 min

Calculate Uncal. Peaks: compound name not specified

Partial Calibration : Yes, identified peaks are recalibrated

Correct All RTs : No, only for identified peaks

Curve Type : Piecewise

Origin : Included

Weight : Equal

```
=====
Injection Date   : 7/24/97 4:02:09 PM           Seq. Line :    2
Sample Name      : .025 ng/ul                   Vial       : 101
Acq. Operator    : sf                           Inj        :    1
                                           Inj Volume : Inj prog
=====
```

```
Sequence File    : C:\HPCHEM\1\SEQUENCE\ALDMETS.S
Acq. Method      : C:\HPCHEM\1\METHODS\CARB.M
Last changed     : 7/24/97 3:58:38 PM by sf
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\CARB.M
Last changed     : 7/24/97 4:25:26 PM by sf
                  (modified after loading)
carbamate method
=====
```

External Standard Report (after recalibration)

Sorted by Retention Time

```
Calib. Data Modified :      Thursday, July 24, 1997 4:25:26 PM
Multiplier           :      1.000000
Dilution             :      1.000000
Uncalibrated Peaks   :      compound name not specified
```

Signal 1: FLD1 A, Ex=330, Em=464

RT [min]	Sig	Type	Area [%?s]	Amt/Area	Amount [ug/spl]	Grp	Name
1.792	1	VV	1.16983	0.00000	0.00000		?
3.134	1	VV	4.12376	0.00000	0.00000		?
3.201	1	VV	9.11323	0.00000	0.00000		?
4.667	1	VV	1.00703	0.00000	0.00000		?
6.480	1	BV	2.67483	3.73855e-1	1.00000		sulfoxide
7.721	1	BV	3.10021	3.22559e-1	1.00000		sulfone
11.729	1	BV	3.70002	2.70269e-1	1.00000		aldicarb
22.025	1	VV	2.87120	0.00000	0.00000		?

```
Totals :                               3.00000
```

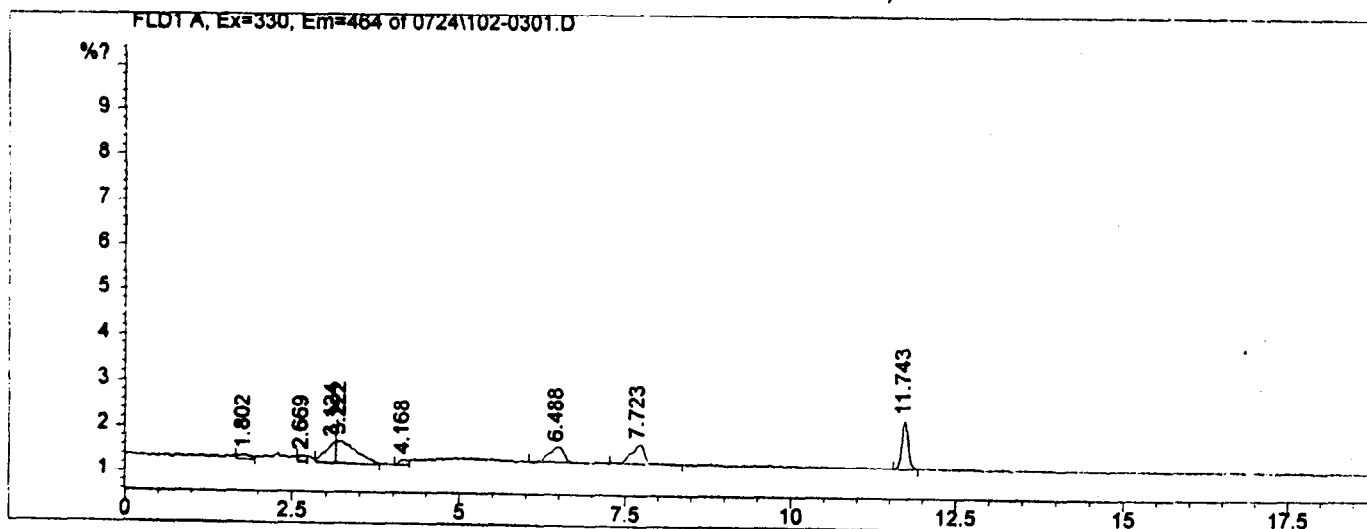
\*\*\* End of Report \*\*\*

Injection Date : 7/24/97 4:29:15 PM Seq. Line : 3  
Sample Name : .05 ng/ul Vial : 102  
Acq. Operator : sf Inj : 1  
Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S  
Acq. Method : C:\HPCHEM\1\METHODS\CARB.M  
Last changed : 7/24/97 4:25:44 PM by sf  
(modified after loading)  
Analysis Method : C:\HPCHEM\1\METHODS\CARB.M  
Last changed : 7/24/97 4:52:35 PM by sf  
(modified after loading)

carbamate method

Instrument Conditions: At Start At Stop  
Temperature: 27.8 27.9 °C  
Pressure: 154.1 155.0 bar  
Flow: 1.000 1.000 ml/min



Calibration Table (after recalibration)

Calib. Data Modified : Thursday, July 24, 1997 4:52:34 PM

Level 2 calibrated: Replace Response Factors,  
No Update of Retention Times

Signal 1 : FLD1 A, Ex=330, Em=464

Injection Date : 7/24/97 4:29:15 PM

Seq. Line : 3

Sample Name : .05 ng/ul

Vial : 102

Acq. Operator : sf

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Acq. Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/24/97 4:25:44 PM by sf

(modified after loading)

Analysis Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/24/97 4:52:35 PM by sf

(modified after loading)

carbamate method

RT [min]	Sig	Lvl	Amount [ug/spl]	Area	Amt/Area	Ref Grp	Name
6.558	1	1	1.00000	2.67483	3.73855e-1		sulfoxide
		2	2.00000	5.02540	3.97978e-1		
		3	4.00000	10.31539	3.87770e-1		
		4	40.00000	105.60493	3.78770e-1		
7.755	1	1	1.00000	3.10021	3.22559e-1		sulfone
		2	2.00000	6.40982	3.12021e-1		
		3	4.00000	11.61346	3.44428e-1		
		4	40.00000	116.78109	3.42521e-1		
11.757	1	1	1.00000	3.70002	2.70269e-1		aldicarb
		2	2.00000	7.51185	2.66246e-1		
		3	4.00000	13.94594	2.86822e-1		
		4	40.00000	139.17429	2.87409e-1		

Rel. Reference Window : 8.000 %

Abs. Reference Window : 0.000 min

Rel. Non-ref. Window : 8.000 %

Abs. Non-ref. Window : 0.000 min

Calculate Uncal. Peaks: compound name not specified

Partial Calibration : Yes, identified peaks are recalibrated

Correct All RTs : No, only for identified peaks

Curve Type : Piecewise

Origin : Included

Weight : Equal

Injection Date : 7/24/97 4:29:15 PM

Seq. Line : 3

Sample Name : .05 ng/ul

Vial : 102

Acq. Operator : sf

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Acq. Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/24/97 4:25:44 PM by sf  
(modified after loading)

Analysis Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/24/97 4:52:35 PM by sf  
(modified after loading)

carbamate method

## External Standard Report (after recalibration)

Sorted by Retention Time

Calib. Data Modified : Thursday, July 24, 1997 4:52:34 PM

Multiplier : 1.000000

Dilution : 1.000000

Uncalibrated Peaks : compound name not specified

Signal 1: FLD1 A, Ex=330, Em=464

RT [min]	Sig	Type	Area [%?*s]	Amt/Area	Amount [ug/spl]	Grp	Name
1.802	1	VV	1.44068	0.00000	0.00000		?
2.669	1	VV	1.04340	0.00000	0.00000		?
3.124	1	VV	4.65020	0.00000	0.00000		?
3.222	1	VV	10.32342	0.00000	0.00000		?
4.168	1	PV	1.02429	0.00000	0.00000		?
6.488	1	BBA	5.02540	3.97978e-1	2.00000		sulfoxide
7.723	1	BV	6.40982	3.12021e-1	2.00000		sulfone
11.743	1	BV	7.51185	2.66246e-1	2.00000		aldicarb
22.042	1	VV	1.71892	0.00000	0.00000		?

Totals : 6.00000

\*\*\* End of Report \*\*\*

Data F

ATTACHMENT D-3

Standard Curve Chromatogram - 0.10 ng/ul

ame: .1 ng

Injection Date : 7/24/97 4:56:25 PM

Seq. Line : 4

Sample Name : .1 ng/ul

Vial : 103

Acq. Operator : sf

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Acq. Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/24/97 4:52:56 PM by sf

(modified after loading)

Analysis Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/24/97 5:19:42 PM by sf

(modified after loading)

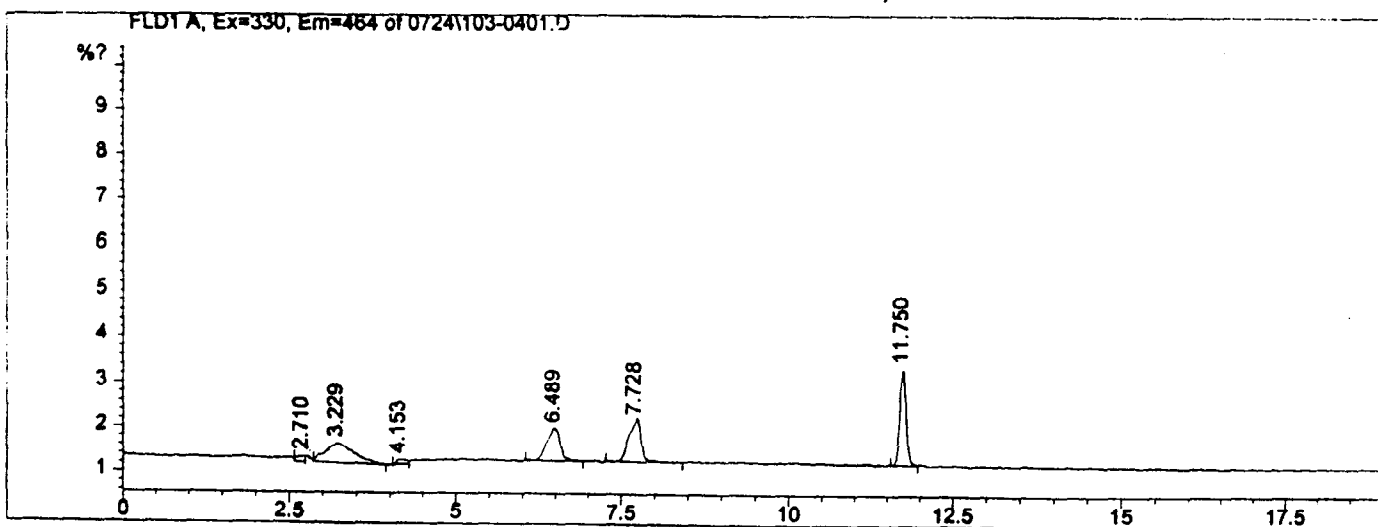
carbamate method

Instrument Conditions: At Start At Stop

Temperature: 27.9 28.0 °C

Pressure: 153.8 154.8 bar

Flow: 1.000 1.000 ml/min



Calibration Table (after recalibration)

Calib. Data Modified : Thursday, July 24, 1997 5:19:42 PM

Level 3 calibrated: Replace Response Factors,  
No Update of Retention Times

Signal 1 : FLD1 A, Ex=330, Em=464

Injection Date : 7/24/97 4:56:25 PM

Seq. Line : 4

Sample Name : .1 ng/ul

Vial : 103

Acq. Operator : sf

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Acq. Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/24/97 4:52:56 PM by sf  
(modified after loading)

Analysis Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/24/97 5:19:42 PM by sf  
(modified after loading)

carbamate method

RT [min]	Sig	Lvl	Amount [ug/spl]	Area	Amt/Area	Ref Grp	Name
6.558	1	1	1.00000	2.67483	3.73855e-1		sulfoxide
		2	2.00000	5.02540	3.97978e-1		
		3	4.00000	10.70984	3.73488e-1		
		4	40.00000	105.60493	3.78770e-1		
7.755	1	1	1.00000	3.10021	3.22559e-1		sulfone
		2	2.00000	6.40982	3.12021e-1		
		3	4.00000	12.35660	3.23714e-1		
		4	40.00000	116.78109	3.42521e-1		
11.757	1	1	1.00000	3.70002	2.70269e-1		aldicarb
		2	2.00000	7.51185	2.66246e-1		
		3	4.00000	14.54610	2.74988e-1		
		4	40.00000	139.17429	2.87409e-1		

Rel. Reference Window : 8.000 %

Abs. Reference Window : 0.000 min

Rel. Non-ref. Window : 8.000 %

Abs. Non-ref. Window : 0.000 min

Calculate Uncal. Peaks: compound name not specified

Partial Calibration : Yes, identified peaks are recalibrated

Correct All RTs : No, only for identified peaks

Curve Type : Piecewise

Origin : Included

Weight : Equal

Injection Date : 7/24/97 4:56:25 PM

Seq. Line : 4

Sample Name : .1 ng/ul

Vial : 103

Acq. Operator : sf

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Acq. Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/24/97 4:52:56 PM by sf  
(modified after loading)

Analysis Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/24/97 5:19:42 PM by sf  
(modified after loading)

carbamate method

## External Standard Report (after recalibration)

## Sorted by Retention Time

Calib. Data Modified : Thursday, July 24, 1997 5:19:42 PM

Multiplier : 1.000000

Dilution : 1.000000

Uncalibrated Peaks : compound name not specified

Signal 1: FLD1 A, Ex=330, Em=464

RT [min]	Sig	Type	Area [%?*s]	Amt/Area	Amount [ug/spl]	Grp	Name
2.710	1	VV	1.08994	0.00000	0.00000		?
3.229	1	VV	13.40528	0.00000	0.00000		?
4.153	1	VV	1.10979	0.00000	0.00000		?
6.489	1	BV	10.70984	3.73488e-1	4.00000		sulfoxide
7.728	1	BV	12.35660	3.23714e-1	4.00000		sulfone
11.750	1	BV	14.54610	2.74988e-1	4.00000		aldicarb
22.030	1	VV	1.52726	0.00000	0.00000		?

Totals :

12.00000

\*\*\* End of Report \*\*\*



## Standard Curve Chromatogram - 1.0 ng/ul

Injection Date : 7/24/97 5:23:27 PM

Seq. Line : 5

Sample Name : 1ng/ul

Vial : 104

Acq. Operator : sf

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Acq. Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/24/97 5:20:00 PM by sf  
(modified after loading)

Analysis Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/24/97 5:46:44 PM by sf  
(modified after loading)

carbamate method

=====

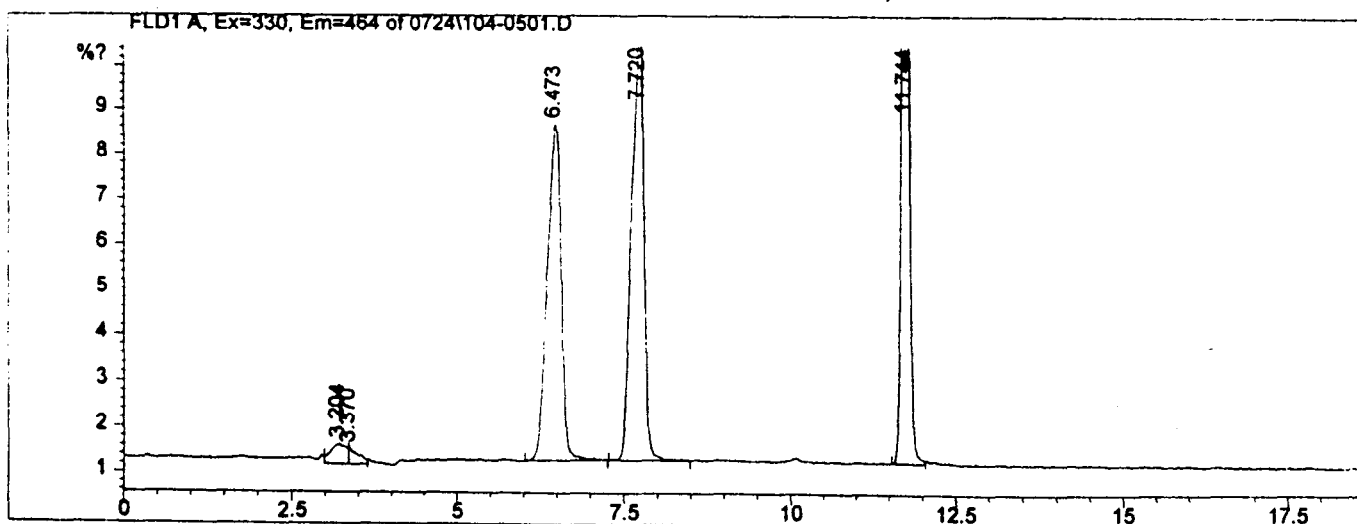
=====

Instrument Conditions: At Start At Stop

Temperature: 28.1 28.2 °C

Pressure: 153.6 155.2 bar

Flow: 1.000 1.000 ml/min



=====

## Calibration Table (after recalibration)

=====

Calib. Data Modified : Thursday, July 24, 1997 5:46:44 PM

Level 4 calibrated: Replace Response Factors,  
No Update of Retention Times

Signal 1 : FLD1 A, Ex=330, Em=464

Injection Date : 7/24/97 5:23:27 PM  
 Sample Name : lng/ul  
 Acq. Operator : sf

Seq. Line : 5  
 Vial : 104  
 Inj : 1  
 Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S  
 Acq. Method : C:\HPCHEM\1\METHODS\CARB.M  
 Last changed : 7/24/97 5:20:00 PM by sf  
 (modified after loading)  
 Analysis Method : C:\HPCHEM\1\METHODS\CARB.M  
 Last changed : 7/24/97 5:46:44 PM by sf  
 (modified after loading)

carbamate method

RT [min]	Sig	Lvl	Amount [ug/spl]	Area	Amt/Area	Ref Grp	Name
6.558	1	1	1.00000	2.67483	3.73855e-1		sulfoxide
		2	2.00000	5.02540	3.97978e-1		
		3	4.00000	10.70984	3.73488e-1		
		4	40.00000	108.33380	3.69229e-1		
7.755	1	1	1.00000	3.10021	3.22559e-1		sulfone
		2	2.00000	6.40982	3.12021e-1		
		3	4.00000	12.35660	3.23714e-1		
		4	40.00000	120.45231	3.32082e-1		
11.757	1	1	1.00000	3.70002	2.70269e-1		aldicarb
		2	2.00000	7.51185	2.66246e-1		
		3	4.00000	14.54610	2.74988e-1		
		4	40.00000	145.03438	2.75797e-1		

Rel. Reference Window : 8.000 %  
 Abs. Reference Window : 0.000 min  
 Rel. Non-ref. Window : 8.000 %  
 Abs. Non-ref. Window : 0.000 min  
 Calculate Uncal. Peaks : compound name not specified  
 Partial Calibration : Yes, identified peaks are recalibrated  
 Correct All RTs : No, only for identified peaks

Curve Type : Piecewise  
 Origin : Included  
 Weight : Equal

```
=====
Injection Date   : 7/24/97 5:23:27 PM           Seq. Line :    5
Sample Name      : lng/ul                       Vial      : 104
Acq. Operator    : sf                          Inj       :    1
                                           Inj Volume : Inj prog
=====
```

```
Sequence File    : C:\HPCHEM\1\SEQUENCE\ALDMETS.S
Acq. Method      : C:\HPCHEM\1\METHODS\CARB.M
Last changed     : 7/24/97 5:20:00 PM by sf
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\CARB.M
Last changed     : 7/24/97 5:46:44 PM by sf
                  (modified after loading)
```

carbamate method

```
=====
External Standard Report (after recalibration)
=====
```

## Sorted by Retention Time

```
Calib. Data Modified :    Thursday, July 24, 1997 5:46:44 PM
Multiplier           :    1.000000
Dilution             :    1.000000
Uncalibrated Peaks   :    compound name not specified
```

Signal 1: FLD1 A, Ex=330, Em=464

RT [min]	Sig	Type	Area [%?*s]	Amt/Area	Amount [ug/spl]	Grp	Name
3.204	1	VV	7.39306	0.00000	0.00000	?	
3.370	1	VV	3.79662	0.00000	0.00000	?	
6.473	1	BBA	108.33380	3.69229e-1	40.00000		sulfoxide
7.720	1	BV	120.45231	3.32082e-1	40.00000		sulfone
11.744	1	BV	145.03438	2.75797e-1	40.00000		aldicarb
22.050	1	VV	2.04170	0.00000	0.00000	?	

```
Totals :                               120.00000
```

```
=====
*** End of Report ***
```

California Department of Food and Agriculture  
Center for Analytical Chemistry  
Worker Health and Safety Laboratory  
3292 Meadowview Road  
Sacramento, CA 95832

Number: WHS-AD-11  
Date: 02/05/96  
Revision:  
Replaces:  
Page: 1 of 3

## STANDARD OPERATING PROCEDURE

**Title:** Data Generation and Reporting

**Purpose:** To Provide a Standardized Procedure for the Generation and Reporting of Chromatographic Data

**Scope:** All laboratory personnel.

### Procedure:

Any conflict with instructions in the method or protocol must be resolved with senior staff, the study director, and documented before proceeding.

The number of standards used should adequately describe the standard curve shape. Typically this is 3-5 points spanning 1-2 orders of magnitude for linear systems. For non-linear systems, additional points or narrower concentration ranges may be needed. Calibration curves should include a data point near the instrument MDL of the compound(s), or a point that approximates the project LOD. All samples with responses higher than the upper limit of the standard curve must be diluted and reanalyzed.

The number and concentration of standards necessary to "adequately describe" the curve shape depend on the type of curve fitting used for data analysis as well as the actual shape of the curve, which in turn depends on the detector used and the chemical being analyzed. In the case of point-to-point curve fitting (used by HP 5880 and 3396 integrators), the number of standards and their concentrations should be chosen so that the maximum quantitative error between a smooth curve and the point-to-point line, measured at the midpoint between consecutive standard levels, is 15% or less. Curve-fit errors in systems that can use quadratic functions (HP MSD, Varian Saturn) are much less, and consequently wider concentration ranges can be used.

In general, using peak heights for GC data will minimize errors because it reduces the effect of small leading or trailing peak interferences. For LC work, peak areas yield better data because of the tendency for LC peaks to widen and shorten during a run due to the effect of developing column voids.

Retention times should be reproducible to better than 1% in most cases for both LC and GC. Capillary GC and gradient LC times should be even better. Some systems will

slowly drift due to changing ambient conditions in the lab, but consecutive runs should show very small changes.

Samples must be run in groups small enough that the standard curves on either side of them will not vary by more than +/- 15%. Sufficient data should be generated during method development to provide guidance for the chemist on this number, and that information should be included in the method. Typically, no more than 10-20 samples should be analyzed between standard curves. 'Conditioning' samples and cooling GC analytical systems between batches may provide more consistent data.

Residues are generally reported in micrograms/sample. In the absence of complicating factors, levels should be reported as follows:

>= 1000 ugs	to nearest 10 ug
100 to 999 ugs	to nearest ug
10 to 99.9 ugs	to nearest 0.1 ug
1 to 9.99 ugs	to nearest 0.01 ug
0.010 to 999 ugs	to nearest 0.001 ug

To prevent confusion when reporting high levels of residue, do not mix reporting units. That is, do not report some values as ugs/sample, and some as mgs/sample within the same group of samples, unless the unit changes are *clearly* marked to draw the reader's attention.

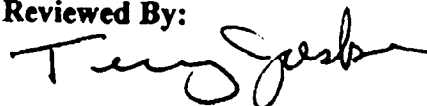
Recovery data should be reported, but sample results NOT corrected for recovery. If corrected results are reported, a notation explicitly stating that fact should be included on the report sheet.

WHS-AD-11

Revision:

Page: 3 of 3

Reviewed By:



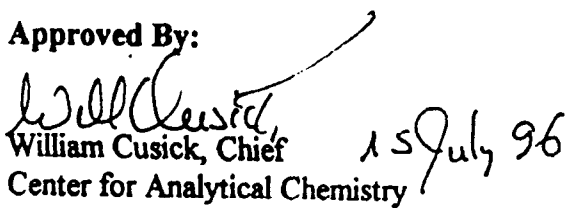
Terry Jackson, QA Officer  
Center for Analytical Chemistry

Approved By:

 7/12/96

Lilia Rivera, Program Supervisor  
Center for Analytical Chemistry

Approved By:

 15 July 96

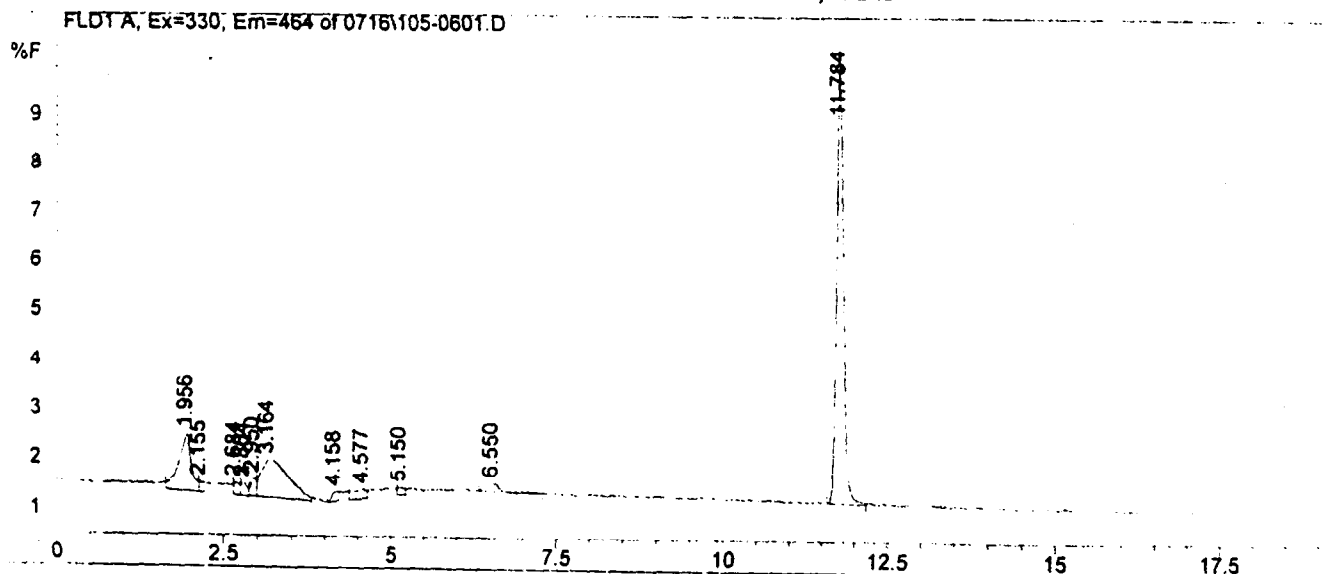
William Cusick, Chief  
Center for Analytical Chemistry

## Resin Lab Spike Chromatogram - 700 ng/spl

Injection Date : 7/16/97 4:18:48 PM Seq. Line : 6  
Sample Name : QA-LS-1dil Vial : 105  
Acq. Operator : cb Inj : 1  
Inj Volume : Inj prog

Acq. Method : C:\HPCHEM\1\METHODS\CARB.M  
Last changed : 7/16/97 4:10:29 PM by cb  
Analysis Method : C:\HPCHEM\1\METHODS\CARB.M  
Last changed : 7/17/97 10:21:14 AM by cb  
(modified after loading)

Instrument Conditions: At Start At Stop  
Temperature: 24.2 24.2 °C  
Pressure: 159.1 161.3 bar  
Flow: 1.000 1.000 ml/min



## External Standard Report

Sorted by Retention Time  
Calib. Data Modified : Thursday, July 17, 1997 9:59:35 AM  
Multiplier : 3.335000e-2  
Dilution : 1.000000 : 2 CB 7-1847  
Uncalibrated Peaks : compound name not specified

Signal 1: FLD1 A, Ex=330, Em=464

Injection Date : 7/16/97 4:18:48 PM  
 Sample Name : QA-LS-1dil  
 Acq. Operator : cb

Seq. Line : 6  
 Vial : 105  
 Inj : 1  
 Inj Volume : Inj prog

Acq. Method : C:\HPCHEM\1\METHODS\CARB.M  
 Last changed : 7/16/97 4:10:29 PM by cb  
 Analysis Method : C:\HPCHEM\1\METHODS\CARB.M  
 Last changed : 7/17/97 10:21:14 AM by cb  
 (modified after loading)

RT [min]	Sig	Type	Area [%F*s]	Amt/Area	Amount [ug/spl]	Grp	Name
1.956	1	VV	14.69682	0.00000	0.00000	?	
2.155	1	VV	1.41390	0.00000	0.00000	?	
2.684	1	VV	1.39944	0.00000	0.00000	?	
2.804	1	VV	1.61221	0.00000	0.00000	?	
2.950	1	VV	1.94610	0.00000	0.00000	?	
3.164	1	VV	21.49023	0.00000	0.00000	?	
4.158	1	PV	1.24534	0.00000	0.00000	?	
4.577	1	VV	2.85748	0.00000	0.00000	?	
5.150	1	VV	1.13571	0.00000	0.00000	?	
6.550	1	PBA	2.11854	3.59774e-1	2.54192e-2		sulfoxide
7.777	1	*	not found	*			sulfone
11.784	1	VV	64.87293	2.60736e-1	5.64106e-1		aldicarb
22.035	1	PV	1.80817	0.00000	0.00000	?	

Totals : 5.89526e-1

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

CB 7-18-97

\*\*\* End of Report \*\*\*

Aldicarb:  $\frac{564 \text{ ng/spl}}{700 \text{ ng spiked}} \times 100 = 80.57\%$



## Resin Trip Spike Chromatogram - 700 ng/spl

Injection Date : 7/16/97 6:24:59 PM

Seq. Line : 10

Sample Name : TS-1dil

Vial : 109

Acq. Operator : cb

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/16/97 4:10:29 PM by cb

Instrument Conditions: At Start At Stop  
Temperature: 24.1 24.3 °C  
Pressure: 157.3 156.0 bar  
Flow: 1.000 1.000 ml/min

FLD1 A, Ex=330, Em=464 of 07161109-1001.D

%F

9

8

7

6

5

4

3

2

1

0

2.5

5

7.5

10

12.5

15

17.5

1.930

2.680

3.168

4.151

4.673

4.933

6.555

11.770

## External Standard Report

Sorted by Retention Time

Calib. Data Modified : Wednesday, July 16, 1997 4:10:29 PM

Multiplier : 6.667000e-2

Dilution : 1.000000 : 4 CB 7-17-97

Uncalibrated Peaks : compound name not specified

Signal 1: FLD1 A, Ex=330, Em=464

Injection Date : 7/16/97 6:24:59 PM

Seq. Line : 10

Sample Name : TS-1dil

Vial : 109

Acq. Operator : cb

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/16/97 4:10:29 PM by cb

RT [min]	Sig	Type	Area [%F*s]	Amt/Area	Amount [ug/sp1]	Grp	Name
1.930	1	VV	7.21614	0.00000	0.00000	?	
2.680	1	VV	1.68195	0.00000	0.00000	?	
3.168	1	VV	15.56869	0.00000	0.00000	?	
4.151	1	PV	1.07101	0.00000	0.00000	?	
4.291	1	VV	1.02937	0.00000	0.00000	?	
4.673	1	VV	1.02780	0.00000	0.00000	?	
4.933	1	VV	1.08988	0.00000	0.00000	?	
6.555	1	BV	3.08353	3.52739e-1	7.25157e-2		sulfoxide
7.755	1		* not found *				sulfone
11.770	1	BV	42.95075	2.61302e-1	7.48246e-1		aldicarb
22.036	1	PV	1.31195	0.00000	0.00000	?	

Totals :

8.20762e-1

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

\*\*\* End of Report \*\*\*

CB 1-18-97

Aldicarb:  $\frac{748 \text{ ng/sp1}}{700 \text{ ng spiked}} \times 100 = 106.86\%$

Aldicarb + Sulfoxide:  $\frac{748 + 64.8 \text{ ng/sp1}}{700 \text{ ng Aldicarb sp1}} \times 100 = 112.11\%$

Sulfoxide: See Chromatogram Sample ID: TS-1 rr 0716\110-1101.D

## ATTACHMENT F-3

Resin Field Spike Chromatogram - 700 ng/spl

WFS1

Injection Date : 7/16/97 8:31:12 PM

Seq. Line : 14

Sample Name : WFS1rr

Vial : 113

Acq. Operator : cb

Inj : 1

Inj Volume : Inj prog

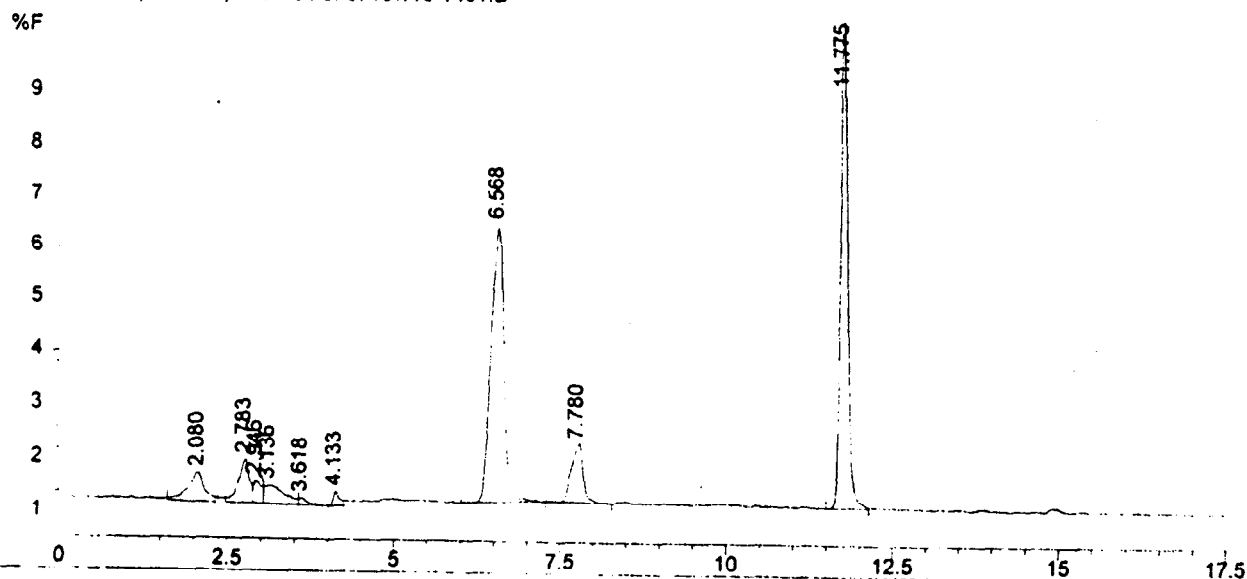
Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/16/97 4:10:29 PM by cb

Instrument Conditions: At Start At Stop  
Temperature: 25.9 26.0 °C  
Pressure: 153.5 156.4 bar  
Flow: 1.000 1.000 ml/min

FLD1 A, Ex=330, Em=464 of 07161113-1401.D



## External Standard Report

Sorted by Retention Time

Calib. Data Modified : Wednesday, July 16, 1997 4:10:29 PM

Multiplier : 1.667000e-2

Dilution : 1.000000

Uncalibrated Peaks : compound name not specified

Signal 1: FLD1 A, Ex=330, Em=464

140

Injection Date : 7/16/97 8:31:12 PM

Seq. Line : 14

Sample Name : WFS1rr

Vial : 113

Acq. Operator : cb

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/16/97 4:10:29 PM by cb

RT [min]	Sig	Type	Area [%F*s]	Amt/Area	Amount [ug/spl]	Grp	Name
2.080	1	VV	9.50730	0.00000	0.00000		?
2.783	1	VV	9.37794	0.00000	0.00000		?
2.946	1	VV	3.47876	0.00000	0.00000		?
3.136	1	VV	7.53400	0.00000	0.00000		?
3.618	1	VV	1.26341	0.00000	0.00000		?
4.133	1	PV	1.77034	0.00000	0.00000		?
6.568	1	BBA	70.07937	3.54367e-1	4.13980e-1		sulfoxide
7.780	1	BV	13.66893	2.78639e-1	6.34909e-2		sulfone
11.775	1	BV	70.66419	2.60646e-1	3.07033e-1		aldicarb

Totals :

7.84504e-1

CB 7-17-97

\*\*\* End of Report \*\*\*

$$\frac{307 \text{ ng/spl Aldicarb} + 63.5 \text{ ng/spl Sulfone} + 414 \text{ ng/spl Sulfoxide}}{700 \text{ ng Aldicarb spiked}} \times 100 = 112.07\%$$

Resin On-Going QC Spike Chromatogram - 50 ng/spl Aldicarb, SO & SO<sub>2</sub> (each)

P: Lab

Injection Date : 7/29/97 12:19:05 AM

Seq. Line : 24

Sample Name : Lab Spike 2

Vial : 123

Acq. Operator : cb

CB 7-29-97

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/29/97 12:15:41 AM by cb

(modified after loading)

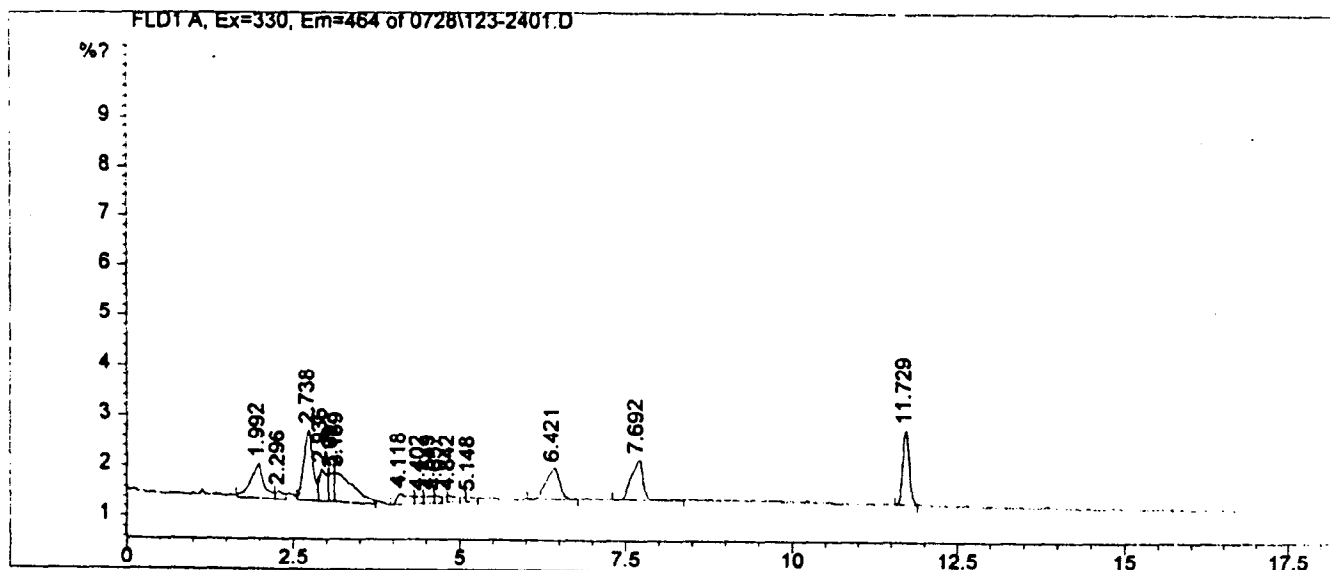
carbamate method

Instrument Conditions: At Start At Stop

Temperature: 28.4 28.3 °C

Pressure: 155.5 157.3 bar

Flow: 1.000 1.000 ml/min



## External Standard Report

Sorted by Retention Time

Calib. Data Modified : Monday, July 28, 1997 4:00:58 PM

Multiplier : 1.667000e-2

Dilution : 1.000000

Uncalibrated Peaks : compound name not specified

Signal 1: FLD1 A, Ex=330, Em=464

Injection Date : 7/29/97 12:19:05 AM

Seq. Line : 24

Sample Name : Lab Spike

Vial : 123

Acq. Operator : cb

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/29/97 12:15:41 AM by cb

(modified after loading)

carbamate method

RT [min]	Sig	Type	Area [%*s]	Amt/Area	Amount [ug/spl]	Grp	Name
1.992	1	VV	9.57038	0.00000	0.00000	?	
2.296	1	VV	1.38980	0.00000	0.00000	?	
2.738	1	VV	13.50778	0.00000	0.00000	?	
2.936	1	VV	5.14743	0.00000	0.00000	?	
3.093	1	VV	3.12747	0.00000	0.00000	?	
3.169	1	VV	12.14086	0.00000	0.00000	?	
4.118	1	PV	3.01058	0.00000	0.00000	?	
4.402	1	VV	1.31653	0.00000	0.00000	?	
4.549	1	VV	1.43042	0.00000	0.00000	?	
4.657	1	VV	1.09848	0.00000	0.00000	?	
4.842	1	VV	1.44463	0.00000	0.00000	?	
5.148	1	VV	1.04210	0.00000	0.00000	?	
6.421	1	BV	9.06302	3.53729e-1	5.34416e-2		sulfoxide
7.692	1	BV	10.40110	3.15731e-1	5.47434e-2		sulfone
11.729	1	BV	10.27557	2.71344e-1	4.64796e-2		aldicarb
21.962	1	PV	2.99277	0.00000	0.00000	?	

Totals :

1.54665e-1

CB 7-29-97

\*\*\* End of Report \*\*\*

Sulfoxide:  $\frac{53.4 \text{ ng recovered}}{50.0 \text{ ng spiked}} \times 100 = 106.80\%$

Sulfone:  $\frac{54.7 \text{ ng recovered}}{50.0 \text{ ng spiked}} \times 100 = 109.40\%$

Aldicarb:  $\frac{46.5 \text{ ng recovered}}{50.0 \text{ ng spiked}} \times 100 = 93.00\%$

143

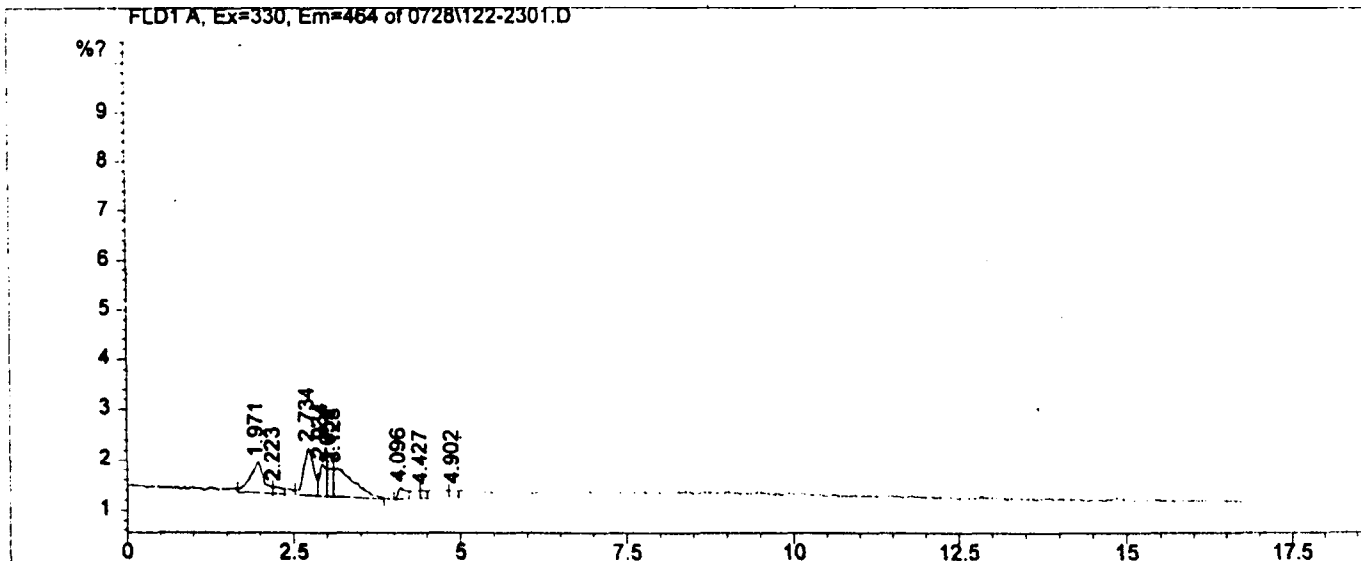
## Resin Blank On-Going QC Chromatogram

Injection Date : 7/28/97 11:52:11 PM Seq. Line : 23  
Sample Name : Lab Blank 2 Vial : 122  
Acq. Operator : cb CB 7-29-97 Inj : 1  
Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S  
Method : C:\HPCHEM\1\METHODS\CARB.M  
Last changed : 7/28/97 11:48:43 PM by cb  
(modified after loading)

carbamate method

Instrument Conditions: At Start At Stop  
Temperature: 28.5 28.4 °C  
Pressure: 155.8 157.4 bar  
Flow: 1.000 1.000 ml/min



## External Standard Report

Sorted by Retention Time

Calib. Data Modified : Monday, July 28, 1997 4:00:58 PM  
Multiplier : 1.667000e-2  
Dilution : 1.000000  
Uncalibrated Peaks : compound name not specified

Signal 1: FLD1 A, Ex=330, Em=464

Injection Date : 7/28/97 11:52:11 PM

Seq. Line : 23

Sample Name : Lab Blank

Vial : 122

Acq. Operator : cb

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/28/97 11:48:43 PM by cb

(modified after loading)

carbamate method

RT [min]	Sig	Type	Area [%*s]	Amt/Area	Amount [ug/spl]	Grp	Name
1.971	1	VV	8.65440	0.00000	0.00000	?	
2.223	1	VV	1.31075	0.00000	0.00000	?	
2.734	1	VV	9.87712	0.00000	0.00000	?	
2.934	1	VV	4.30984	0.00000	0.00000	?	
3.055	1	VV	3.06299	0.00000	0.00000	?	
3.125	1	VV	12.09978	0.00000	0.00000	?	
4.096	1	PV	1.92149	0.00000	0.00000	?	
4.427	1	VV	1.07458	0.00000	0.00000	?	
4.902	1	VV	1.09158	0.00000	0.00000	?	
6.558	1	*	not found	*			sulfoxide
7.755	1	*	not found	*			sulfone
11.757	1	*	not found	*			aldicarb
21.954	1	PV	2.65767	0.00000	0.00000	?	

Totals :

0.00000

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

## Area Percent Report

Sorted by Retention Time

Calib. Data Modified : Monday, July 28, 1997 4:00:58 PM

Multiplier : 1.667000e-2

Dilution : 1.000000

Signal 1: FLD1 A, Ex=330, Em=464



Injection Date : 7/28/97 11:52:11 PM

Seq. Line : 23

Sample Name : Lab Blank

Vial : 122

Acq. Operator : cb

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/28/97 11:48:43 PM by cb

(modified after loading)

carbamate method

Peak #	RT [min]	Sig	Type	Area [%?*s]	Area %	Name
1	1.971	1	VV	8.65440	3.13e-1	?
2	2.223	1	VV	1.31075	4.74e-2	?
3	2.734	1	VV	9.87712	3.57e-1	?
4	2.934	1	VV	4.30984	1.56e-1	?
5	3.055	1	VV	3.06299	1.11e-1	?
6	3.125	1	VV	12.09978	4.38e-1	?
7	4.096	1	PV	1.92149	6.95e-2	?
8	4.427	1	VV	1.07458	3.89e-2	?
9	4.902	1	VV	1.09158	3.95e-2	?
10	21.954	1	PV	2.65767	9.62e-2	?

Totals : 46.06018

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

\*\*\* End of Report \*\*\*

## ARB Resin Sample Chromatogram - N6

Injection Date : 7/28/97 11:25:13 PM

Seq. Line : 22

Sample Name : 294-N6

Vial : 121

Acq. Operator : cb

Inj : 1

Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S

Method : C:\HPCHEM\1\METHODS\CARB.M

Last changed : 7/28/97 11:21:43 PM by cb

(modified after loading)

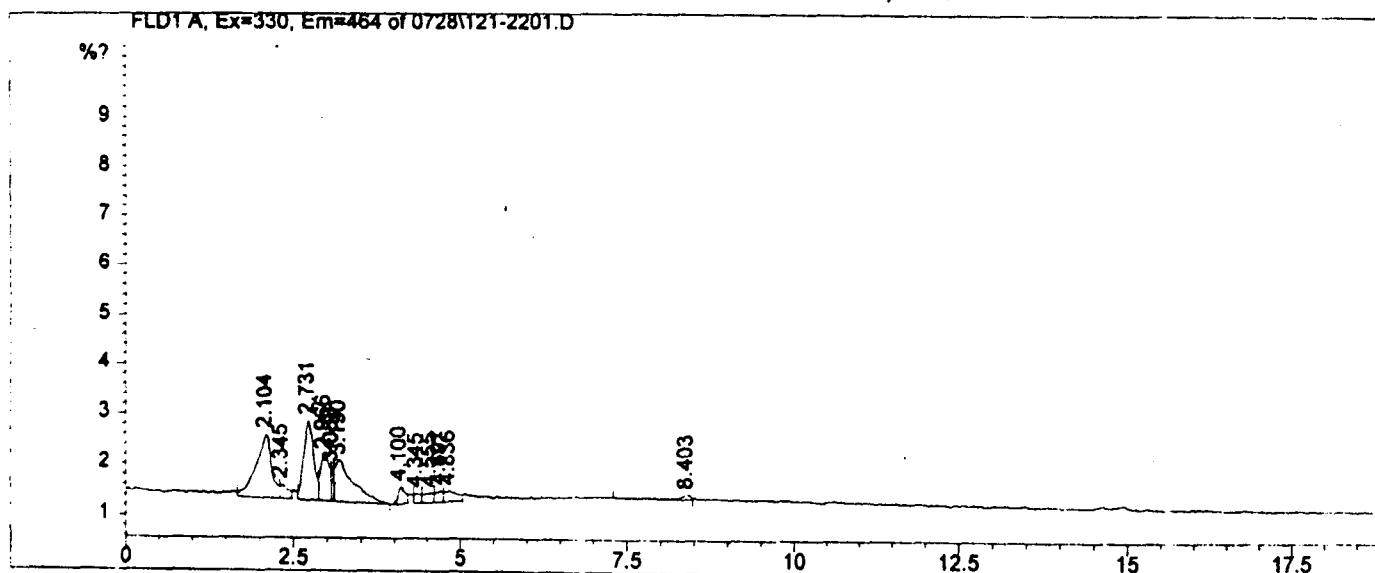
carbamate method

Instrument Conditions: At Start At Stop

Temperature: 28.7 28.6 °C

Pressure: 154.7 156.7 bar

Flow: 1.000 1.000 ml/min



## External Standard Report

Sorted by Retention Time

Calib. Data Modified : Monday, July 28, 1997 4:00:58 PM

Multiplier : 1.667000e-2

Dilution : 1.000000

Uncalibrated Peaks : compound name not specified

Signal 1: FLD1 A, Ex=330, Em=464

=====  
Injection Date : 7/28/97 11:25:13 PM  
Sample Name : 294-N6  
Acq. Operator : cb

Seq. Line : 22  
Vial : 121  
Inj : 1  
Inj Volume : Inj prog

Sequence File : C:\HPCHEM\1\SEQUENCE\ALDMETS.S  
Method : C:\HPCHEM\1\METHODS\CARB.M  
Last changed : 7/28/97 11:21:43 PM by cb  
(modified after loading)

carbamate method  
=====

RT [min]	Sig	Type	Area [%?s]	Amt/Area	Amount [ug/spl]	Grp	Name
2.104	1	VV	19.97105	0.00000	0.00000	?	
2.345	1	VV	2.29746	0.00000	0.00000	?	
2.731	1	VV	15.97599	0.00000	0.00000	?	
2.966	1	VV	8.05523	0.00000	0.00000	?	
3.083	1	VV	1.70558	0.00000	0.00000	?	
3.190	1	VV	15.87574	0.00000	0.00000	?	
4.100	1	PV	2.51331	0.00000	0.00000	?	
4.345	1	VV	1.37205	0.00000	0.00000	?	
4.555	1	VV	2.00428	0.00000	0.00000	?	
4.697	1	VV	1.77667	0.00000	0.00000	?	
4.836	1	VV	3.02459	0.00000	0.00000	?	
6.558	1	*	not found	*			sulfoxide
7.755	1	*	not found	*			sulfone
8.403	1	BV	1.62840	0.00000	0.00000	?	
11.757	1	*	not found	*			aldicarb
21.966	1	VV	2.49042	0.00000	0.00000	?	

Totals : 0.00000

1 Warnings or Errors :

. Warning : Calibrated compound(s) not found  
=====

```
=====
Injection Date   : 7/28/97 11:25:13 PM      Seq. Line :   22
Sample Name      : 294-N6                   Vial       :  121
Acq. Operator    : cb                      Inj        :    1
                                           Inj Volume : Inj prog
=====
```

```
Sequence File    : C:\HPCHEM\1\SEQUENCE\ALDMETS.S
Method           : C:\HPCHEM\1\METHODS\CARB.M
Last changed     : 7/28/97 11:21:43 PM by cb
                  (modified after loading)
```

carbamate method

```
=====
                          Area Percent Report
=====
```

Sorted by Retention Time

```
Calib. Data Modified :      Monday, July 28, 1997 4:00:58 PM
Multiplier           :      1.667000e-2
Dilution             :      1.000000
```

Signal 1: FLD1 A, Ex=330, Em=464

Peak #	RT [min]	Sig	Type	Area [%*s]	Area %	Name
1	2.104	1	VV	19.97105	4.23e-1	?
2	2.345	1	VV	2.29746	4.87e-2	?
3	2.731	1	VV	15.97599	3.38e-1	?
4	2.966	1	VV	8.05523	1.71e-1	?
5	3.083	1	VV	1.70558	3.61e-2	?
6	3.190	1	VV	15.87574	3.36e-1	?
7	4.100	1	PV	2.51331	5.32e-2	?
8	4.345	1	VV	1.37205	2.91e-2	?
9	4.555	1	VV	2.00428	4.25e-2	?
10	4.697	1	VV	1.77667	3.76e-2	?
11	4.836	1	VV	3.02459	6.41e-2	?
12	8.403	1	BV	1.62840	3.45e-2	?
13	21.966	1	VV	2.49042	5.28e-2	?

Totals : 78.69077

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

```
=====
*** End of Report ***
```

149

APPENDIX III  
PESTICIDE USE  
REPORTS

1997 PESTICIDE USE REPORT ENTRY/EDIT SCREEN  
Production Agriculture

DCNUM: 172201 GROWER #: 1030773 GROWER: TERRANOVA RANCH INC

TE ID # 365 PLANTED TOTAL 68.0 A S.03 1.16 R.18 MAP-ID 01  
COMMODITY COTTON SITE LOCATION E 1/2 OF NEQ

APPLICATOR TERRANOVA RANCH INC

App Type G

App Date	M	Tr	Area	U	EPA Reg. Number	Amt Used	U	Product Name
05/24/97	G		68.0	A	204- 426-AA-	264	408.00	LB TEMIK BRAND 15G ALD

Chem. Mfg. RHONE-POULFNC AG COMPANY

>-----<-----  
S=Save - D=Delete - TAB=Find - Q=HELP - A=Clear Screen - ESC=Quit G

1997 PESTICIDE USE REPORT ENTRY/EDIT SCREEN  
Production Agriculture

DOCNUM: 0533732 GROWER #: 1030773 GROWER: TERRANOVA RANCH INC

ITE ID # 347 PLANTED TOTAL 72.0 A S.09 T.16 R.18 MAP-ID 01  
COMMODITY COTTON SITE LOCATION W 1/2 OF NWQ

APPLICATOR HELM FERTILIZER

App Date	M	Tr	Area	U	EPA Reg. Number	Amt Used	U	Product Name
06/25/97	G		72.0	A	264- 330-AA-	264	1002.00	LB TEMIK 15% GRANULAR

App-Type P

Chem. Mfg. RHONE-POULENC AG COMPANY

<----->  
F5=Save - F0=Delete - TAB=Find - FQ=HELP - FA=Clear Screen - ESC=Quit G

1997 PESTICIDE USE REPORT ENTRY/EDIT SCREEN  
Production Agriculture

DOC #: 172220 GROWER #: 1030773 GROWER: TERRANOVA RANCH INC

ITE ID # 365 PLANTED TOTAL 68.5 A S.03 T.16 R.18 MAP-ID 01  
COMMODITY COTTON SITE LOCATION W 1/2 OF SWQ

APPLICATOR TERRANOVA RANCH INC

App Date	M	Tr	Area	U	EPA Reg. Number	Amt Used	U	Product Name
03/24/97	G		68.5	A	264- 426-AA-	264	411.00	LB TEMIK BRAND 15% ALD

App-Type G

Chem. Mfg. RHONE-POULENC AG COMPANY

<----->  
F5=Save - F0=Delete - TAB=Find - FQ=HELP - FA=Clear Screen - ESC=Quit G

APPENDIX IV  
DPR's  
MONITORING RECOMMENDATIONS FOR ALDICARB



# Memorandum

George Lew, Chief  
Engineering and Laboratory Branch  
Monitoring and Laboratory Division  
Air Resources Board  
600 North Market Boulevard  
Sacramento, California 95812

Date : April 3, 1996

Place :

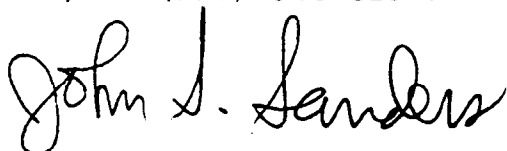
From Department of Pesticide Regulation - 1020 N Street, Room 161  
Sacramento, California 95814-5624

Subject MONITORING RECOMMENDATION FOR ALDICARB

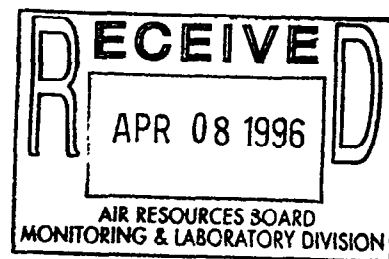
Attached is the Department of Pesticide Regulation's recommendation for monitoring the systemic insecticide aldicarb. This recommendation is provided pursuant to the requirements of AB 1807/3219 (Food and Agricultural Code, Division 7, Chapter 3, Article 1.5). As you know, monitoring recommendations are made using historical use information for the pesticide in question. For this reason, it is essential that the agricultural commissioner, in the county or counties where monitoring will be conducted, be consulted prior to the onset of air monitoring.

We anticipate submission of air monitoring data by October 1997:

If you have any questions please contact Kevin Kelley, of my staff, at (916) 324-4187.



John S. Sanders, Chief  
Environmental Monitoring and  
Pest Management Branch  
(916) 324-4100



Attachment

cc: Paul H. Gosselin, Ronald J. Oshima, Kevin Kelley,  
Madeline Brattesani, Charles M. Andrews, Gary Patterson, Barry Cortez,  
John Donahue - DPR; Genevieve Shiroma, Don Fitzell, Cara Roderick - ARB  
Ted Davis, Agricultural Commissioner Kern County  
M. Hugh Handley, Agricultural Commissioner Kings County



Staff Report

**USE INFORMATION AND AIR MONITORING  
RECOMMENDATION FOR THE PESTICIDAL ACTIVE  
INGREDIENT ALDICARB**

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Principal Author

**Pamela Wales**  
Environmental Research Assistant

State of California  
Department of Pesticide Regulation  
1020 N Street  
Sacramento, California 95814-5624

## USE INFORMATION AND AIR MONITORING RECOMMENDATION FOR THE PESTICIDAL ACTIVE INGREDIENT ALDICARB

### BACKGROUND

To fulfill the requirements of AB 1807/3219 (Food and Agricultural Code, Division 7, Chapter 3, Article 1.5), the Department of Pesticide Regulation (DPR) has previously requested that the Air Resources Board (ARB) document the airborne concentrations of the pesticide aldicarb (2-methyl-2-[methylthio]propionaldehyde *O*-methylcarbamoyloxime). This recommendation provides background and recent use information on aldicarb-containing products, and identifies how they are used.

Aldicarb (CAS: 116-06-3) is a colorless crystalline solid with a faint sulfurous odor. Aldicarb has a molecular formula of  $C_7H_{14}N_2O_2S$ , a molecular weight of 190.27, and a specific density of 1.195 at 25°C. It has a water solubility of 6.0 g/L at 25°C, a Henry's Constant of  $1.45 \times 10^{-9}$  atm·m<sup>3</sup>/mol at 20–25°C, and a vapor pressure of  $3.47 \times 10^{-5}$  mmHg at 25°C. Aldicarb is miscible with most organic solvents.

In soil, aldicarb is rapidly converted to aldicarb sulfoxide in the presence of oxidizing agents and microorganisms. Further oxidation to the sulfone by microorganisms occurs at a slower rate. Mineralization was more rapid in aerobic surface soils than either aerobic or anaerobic subsurface soils; aldicarb degrades rapidly in aerobic silty clay loam soils releasing carbon dioxide. Reported soil half-life ( $t_{1/2}$ ) ranges from 7 to 70 days. In water, the reported hydrolysis half-lives range from 175 to 245 days. In plants, with the exception of cotton, aldicarb is rapidly metabolized to aldicarb sulfoxide, sulfone, and water-soluble noncarbamate compounds. Aldicarb sulfoxide is highly soluble, acts systemically on the plant, and is 10-20 times more active as a cholinesterase inhibitor than the parent compound.

The acute oral LD<sub>50</sub> of aldicarb for rats ranges from 650 to 930 µg/kg. The LC<sub>50</sub> (96 hour) of aldicarb for rainbow trout is 0.88 mg/L, and for bluegill sunfish is 1.5 mg/L. The LC<sub>50</sub> (72 hour) for bluegill sunfish is 100 µg/L. Aldicarb has entered the risk assessment process at DPR under the Birth Defect Prevention Act of 1984 (SB950) primarily because of its cholinesterase inhibition.

### USE OF ALDICARB

As of March 4, 1996, there were four active registrations for products containing aldicarb, all of which are agricultural products. Aldicarb is a soil applied systemic insecticide absorbed via the roots and translocated throughout the plant, providing control of insects and nematodes. Aldicarb is formulated as either granules or flakes. The Signal Word found on these aldicarb-containing products is "Poison/Danger".

Aldicarb use for 1993, 1992 and 1991 is summarized in Table 1, showing aldicarb use by year; and in Table 2, showing aldicarb applications in Kern and Kings Counties. The agricultural use of aldicarb in the six counties listed in Table 1 accounts for 77% to 86% of the total annual aldicarb use. All the remaining 14% to 23% of the total use in California is applied agriculturally in counties not listed in Table 1. Aldicarb, due to its ability to move through porous sandy soils, is not registered for use in Humboldt and Del Norte Counties.

**Table 1. Aldicarb Use by Year (Pounds of Active Ingredient)**

County	1993	1992	1991
Kern	57,198	55,530	54,858
Kings	49,412	32,271	44,864
Tulare	29,194	27,975	22,364
Fresno	50,372	26,618	23,570
County Totals	188,169	144,386	147,647
<i>Percent of Total</i>	<i>79.2%</i>	<i>85.7%</i>	<i>76.6%</i>
<b>CALIFORNIA</b>			
<b>TOTAL</b>	<b>237,734</b>	<b>168,569</b>	<b>192,763</b>

The Pesticide Use Report data summarized in Table 1 show that Kern, Kings, Tulare, and Fresno Counties routinely receive the greatest applications of aldicarb. Yearly applications of aldicarb are consistently largest in Kern County followed by yearly applications in Kings and Tulare Counties. Table 2 shows monthly aldicarb use in Kern, Kings, and Fresno Counties. In 1993, annual aldicarb use in Fresno County nearly doubled when compared to previous years. Nearly all of the increase can be attributed to a three-fold increase in use during the month of April 1993 (Table 2). Because the 1994 use data is not yet available, it cannot be determined if the increased use in Fresno County during April is a trend or one-time occurrence.

**Table 2. Annual aldicarb use by month for Kern, Kings, and Fresno Counties in pounds of active ingredient, during seasons of use.****-Kern County-**

Month	1993	1992	1991
March	1,634	1,915	-
April	21,022	17,518	24,992
May	8,837	12,064	4,424
June	22,441	21,196	20,790
July	1,640	2,837	4,652
August	1,144	-	-
September	479	-	-
<i>Total</i>	<i>57,198</i>	<i>55,530</i>	<i>54,858</i>

**-Kings County-**

Month	1993	1992	1991
March	-	934	186
April	37,834	24,154	29,375
May	8,558	3,496	8,306
June	3,050	3,687	6,922
July	-	-	75
<i>Total</i>	<i>49,442</i>	<i>32,271</i>	<i>44,864</i>

**-Fresno County-**

Month	1993	1992	1991
March	3	289	-
April	41,917	15,103	19,329
May	4,021	6,001	557
June	3,876	5,224	3,044
July	465	-	190
August	-	-	450
<i>Total</i>	<i>50,372</i>	<i>26,617</i>	<i>23,570</i>

Aldicarb is a soil-applied systemic insecticide, acaricide, and nematocide. Most of the aldicarb used in California is applied to cotton, primarily for the control of aphids and thrips.

Additionally, aldicarb has a putative growth enhancing effect on cotton, and for this reason many growers make routine, low-concentration applications (at about 1 lb/acre) in the spring. Table 3 summarizes the total amounts and rates of aldicarb applied to cotton in Kern and Kings Counties in 1993, during the seasons of highest use. 1991 and 1992 use patterns mirror the patterns shown in 1993. It is important to note that each year the rates of aldicarb use on cotton during the month of May changed dramatically. The rate of use doubled during the second half of the month. Each year, this change in use occurred consistently on or about the 15th of May.

**Table 3. 1993 Aldicarb applications to cotton in Kern and Kings Counties during months of highest rate of use, indicating pounds of active ingredient (AI) applied, and average monthly application rates.**

	May 1-15		May 16-31		June 1-15		June 16-30	
County	Lbs Used	Rate	Lbs Used	Rate	Lbs Used	Rate	Lbs Used	Rate
Kern	2,016	1.3	6,748	2.1	14,963	2.0	7,233	1.9
Kings	7,815	0.8	743	2.2	1,719	2.1	1,331	2.2

## RECOMMENDATIONS

### *Ambient Air Monitoring.*

The use patterns for aldicarb suggest that monitoring should occur over a 30- to 45-day sampling period in either Kern or Kings County. In 1993 a notable increase in aldicarb use occurred in Fresno County, indicating that county could be considered, as well, although the 1994 use data should be consulted prior to the onset of sampling. Sampling may be conducted during the month of April in Kings or Fresno Counties; alternatively, sampling may be conducted in Kern County during June. Three to five sampling sites should be selected in relatively high-population areas or in areas frequented by people. In any case, sampling sites should be located near cotton growing areas. Ambient samples should not be collected from samplers immediately adjacent to fields where aldicarb is being applied. At each site, twenty to thirty discrete 24-hour samples should be taken during the sampling period. Background samples should be collected in an area distant to aldicarb applications.

Replicate (co-located) samples are needed for five dates at each sampling location. Two co-located samplers (in addition to the primary sampler) should be run on those days. The date chosen for replicate samples should be distributed over the entire sampling period. They may,

but need not be, the same dates at every site. Field blank and spike samples should be collected at the same environmental (temperature, humidity, exposure to sunlight) and experimental (air flow rates) conditions as those occurring at the time of ambient sampling.

### ***Monitoring of an Application Site.***

The use patterns for aldicarb (Table 3) suggest that application-site monitoring should be collected during the latter half of May through June, that monitoring be conducted in Kern or Kings County, and that monitoring should be associated with applications to cotton. Annually, application rates to cotton generally range from 0.5 to 2.0 pounds of active ingredient per acre. Therefore, monitoring should be conducted in Kern or Kings Counties from mid-May through June and should be related to applications at the highest rate. Aldicarb is extensively applied during this period so care should be taken so that nearby applications do not contaminate collected samples.

A three day monitoring period should be established with sampling times as follows: Application + 1 hour, followed by one 2-hour sample, one 4-hour sample, two 8-hour samples and two 24-hour samples. A minimum of four samplers should be positioned, one on each side of the field. A fifth sampler should be co-located at one position. Since aldicarb is extensively used in the area, background samples should collect enough volume (either 12 hours at 15 liters/minute, or a shorter period with a higher volume pump) to permit a reasonable minimum detection level. Ideally, samplers should be placed a minimum of 20 meters from the field. Field blank and field spike samples should be collected at the same environmental (temperature humidity, exposure to sunlight) and experimental (similar air flow rates) conditions as those occurring at the time of sampling.

We also request that you provide in the monitoring report: 1) An accurate record of the positions of the monitoring equipment with respect to the field, including the distance each monitor is positioned away from the edge of the field, 2) an accurate drawing of the monitoring site showing the precise location of the meteorological equipment, trees, buildings, and other obstacles, 3) meteorological data collected at a minimum of 15-minute intervals including wind speed and direction, humidity, and comments regarding degree of cloud cover, 4) the elevation of each sampling station with respect to the field, and 6) the orientation of the field with respect to North (identified as either true or magnetic North).

APPENDIX V

APPLICATION AND AMBIENT FIELD LOG SHEETS



No background  
FS<sup>2</sup> taken on day #2 → except at Met Station

# LOG BOOK

Project: Aldicarb Application #1

Project #: C97-007

Log Number	Sample ID	Date	Time	Comments	weather o = overcast pc = partly cloudy k = clear   taken by	
1	ALBW1	3/24/97	12:25		K	KEM
		3/24/97	20:45			
2	ALAN1	3/24/97	12:30			
		3/24/97	20:40			
3	ALBE1	3/24/97	13:25			
		3/24/97	20:30			
4	ALBE1A	3/24/97	13:25			
		3/24/97	20:30			
5	ALBS1	3/24/97	12:40			
		3/24/97	20:25			
6	ALBE1	3/24/97	12:55	at Met Station		
		3/24/97	20:50			
7	ALBS2	3/24/97	20:25			
		3/25/97	8:06			
8	ALBE2	3/24/97	20:30			
		3/25/97	8:05			
9	ALBE2D	3/24/97	20:30			
		3/25/97	8:05			
10	ALBN2	3/24/97	20:10			
		3/25/97	8:15			
11	ALBN2	3/24/97	20:15			
		3/25/97	8:25			
12	ALBE2	3/24/97	20:50			
		3/25/97	8:35			
13	ALBS3	3/25/97	8:00			
		3/26/97	9:12			
14	ALBE3	3/25/97	8:05			
		3/26/97	9:18			
15	ALBE3D	3/25/97	8:05			
		3/26/97	9:18			
16	ALBN3	3/25/97	8:15	Pump off when ... plug loose		
		3/25/97	9:25			
17	ALBW3	3/25/97	8:25			
		3/26/97	9:32			
18	ALBA =	3/25/97	8:35			
		3/26/97	9:50			
19	FS154	3/26/97	9:12	<del>at background</del>		
		3/27	5:20			
20	ALOS4	3/26/97	9:12			
		3/27	5:20			
<del>21</del>	<del>FS24</del>	<del>3/26/97</del>	<del></del>			
21	ALOE4	3/26/97	9:18			
		3/27/97	8:30			

Project: Aldicarb Application  
Project #: C97-007

160

# LOG BOOK

Project: Aldicarb Application - #2  
Project #: C97-007

Log Number	Sample ID	Date	Time	Comments	weather o = overcast pc = partly cloudy k = clear   taken by	
1	WB	6/24	0825	back ground	K	HEM
		6/25	0725			
2	WFS1	6/24	0825	field spike		
		6/25	0725			
3	SB	6/24	0825	Pump failure - unplug #11 is it better? dig a hole - back		
		6/25	0728			
4	SFS2	6/24	0835			
		6/25	0728			
5	ER	6/24	0845			
		6/25	0736			
6	EF3	6/24	0845			
		6/25	0736			
7	NR	6/24	0855	Pump problem / fixed chart		
		6/24	1925			
8	NFS3	6/24	0855			
		6/25	0745			
9	W1	6/25	0725			
		6/25	1820			
10	S1	6/25	0728			
		6/25	1805			
11	S10	6/25	0728			
		6/25	1805			
12	E1	6/25	0736			
		6/25	1810			
13	N1	6/25	0745			
		6/25	1813			
14	W2	6/25	1810			
		6/25	1955			
15	S2	6/25	1805			
		6/25	2000			
16	S2A	6/25	1805			
		6/25	2000			
17	E2	6/25	1810			
		6/25	2005			
18	N2	6/25	1813			
		6/25	2010			
19	W3	6/25	1955			
		6/26	0001			
20	S3	6/25	2000			
		6/26	0003			
21	S30	6/25	2000			
		6/26	0003			
22	E3	6/25	2005			
		6/26	0008			
23	N3	6/25	2010			
		6/26	0014			

**LOG BOOK**  
Project: Aldicarb Application  
Project #: C97-007

Log Number	Sample ID	Date	Time	Comments	weather o = overcast pc = partly cloudy k = clear   taken by	
24	W4	6/26	0001		K	KEM
		6/26	0810			
25	S4	6/26	2000			
		6/26	2015			
26	S4C	6/26	2000			
		6/26	0815			
27	E4	6/26	2005			
		6/26	0820			
28	N4	6/26	2010			
		6/26	0825			✓
29	W5	6/26	0810			
		6/27	0840			
30	S5	6/26	0815			
		6/27	0850			
31	S5C	6/26	0815			
		6/27	0850			
32	E5	6/26	0820			
		6/27	0855			
33	N5	6/26	0825			
		6/27	0900		↓	↓
34	TS1	6/27	0845	Top spike		
35	TS2	6/27	0845			
36	TS3	6/27	0845			
37	TS4	6/27	0845			
38	B1	6/27	0845	Blank		
39	W6	6/27	0845			
		6/28	0930			
40	S6	6/27	0850			
		6/28	0930			
41	S6D	6/27	0850			
		6/28	0930			
42	E6	6/27	0855			
		6/28	0930			
43	N6	6/27	0900			
		6/28	0935			

**LOG BOOK**  
 Project: Aldicarb Ambient - *Fresno*  
 Project #: C97-004

Log Number	Sample ID	Date	Time	Comments	weather o = overcast pc = partly cloudy k = clear   taken by	
1	SJ-1	3/24/97 3/25/97	1130 1100	PRIMARY ROTOMETER IS 3A	K	H.J.
2	HEL-1	3/24/97 3/25/97	1215 140	" " 4A		
3	FP-1	3/24/97 3/25/97	1235 1200	" " 5A		
4	BOR-1	3/24/97 3/25/97	1330 1245	" " 2A		
5	ARB-1	3/24/97 3/25/97	1445 1430	" " 6A		
6	SJ-2	3/25/97 3/26/97	1100 0900			
7	HEL-2	3/25/97 3/26/97	1140 0935			
8	FP-2	3/25/97 3/26/97	1205 1005			
9	BOR-2	3/25/97 3/26/97	1245 1035			
10	ARB-2	3/25/97 3/26/97	1430 1405			
11	SJ-3	3/26/97 3/27/97	0900 0930			
12	SJ-3D	3/26/97 3/27/97	0900 0830	DUPLICATE ROTOMETER IS 3B		
13	HEL-3	3/26/97 3/27/97	0935 0845			
14	HEL-3D	3/26/97 3/27/97	0935 0845	" " 4B		
15	FP-3	3/26/97 3/27/97	1005 0915			
16	FP-3D	3/26/97 3/27/97	1005 0915	" " 5B		
17	BOR-3	3/26/97 3/27/97	1035 1000			
18	BOR-3D	3/26/97 3/27/97	1035 1000	" " 2B		
19	ARB-3	3/26/97 3/27/97	1405 1200			
20	ARB-3D	3/26/97 3/27/97	1405 1200	" " 6B		
21	B-3	3/26/97	1405	Blank DONE AT ARB SITE		
22	SJ-4	3/27/97 3/28/97	0830 0830			

**LOG BOOK**  
Project: Aldicarb Ambient  
Project #: C97-004

Log Number	Sample ID	Date	Time	Comments	weather o = overcast pc = partly cloudy k = clear   taken by	
23	HEL-4	3/27/97	0845		K	L.J.
		3/28/97	0900			
24	FP-4	3/27/97	0915		↓	↓
		3/28/97	0920			
25	BOR-4	3/27/97	1000		↓	↓
		3/28/97	1000			
26	ARB-4	3/27/97	1200	Sample was collected FIRST ON 3/28/97	↓	↓
		3/28/97	0730			
27	SJ-5	3/31/97	1000		NORTH 20 MPH	LDT
		4-1-97	0930			
28	HEL-5	3-31-97	1030		↓	
		4-1-97	1000			
29	FP-5	3-31-97	1100		↓	
		4-1-97	1025			
30	BOR-5	3-31-97	1130		↓	
		4-1-97	1050			
31	ARB-5	3-31-97	1330		↓	
		4-1-97	1245			
32	SJ-6	4-1-97	0930		NORTH 35 MPH	
		4-2-97	0915			
33	HEL-6	4-1-97	1000		K	
		4-2-97	0945			
34	FP-6	4-1-97	1025		↓	
		4-2-97	1010			
35	BOR-6	4-1-97	1050		↓	
		4-2-97	1100			
36	ARB-6	4-1-97	1245		↓	
		4-2-97	1335			
37	SJ-7	4-2-97	0915		↓	
		4-3-97	0900			
38	SJ-7D	4-2-97	0915		↓	
		4-3-97	0900			
39	HEL-7	4-2-97	0945		↓	
		4-3-97	1000			
40	HEL-7D	4-2-97	0945		↓	
		4-3-97	1000			
41	FP-7	4-2-97	1010		↓	
		4-3-97	1025			
42	FP-7D	4-2-97	1010		↓	
		4-3-97	1025			
43	BOR-7	4-2-97	1100		↓	
		4-3-97	1130			
44	BOR-7D	4-2-97	1100		↓	
		4-3-97	1130			

**LOG BOOK**  
Project: Aldicarb Ambient  
Project #: C97-004

Log Number	Sample ID	Date	Time	Comments	weather o = overcast pc = partly cloudy k = clear   taken by	
45	ARB-7	4-2-97	1335		K	LOT
		4-3-97	1330			
46	ARB-8	4-2-97	1335			
		4-3-97	1330			
47	BLAN-7	4-2-97	1100	Barrel School		
48	SS-8	4-3-97	0900			
		4-4-97	0830			
49	HEL-8	4-3-97	1000			
		4-4-97	0905			
50	FP-8	4-3-97	1025			
		4-4-97	0930			
51	BOR-8	4-3-97	1130			
		4-4-97	1000			
52	ARB-8	4-3-97	1330		V	V
		4-4-97	1200			
53	SS-9	4-7-97	0800		K	DR
		4-8-97	0930			
54	HEL-9	4-7-97	0815			
		4-8-97	0930			
55	FP-9	4-7-97	0830			
		4-8-97	1010			
56	BOR-9	4-7-97	0900			
		4-8-97	1035			
57	ARB-9	4-7-97	1110			
		4-8-97	1230			
58	SS-10	4-8-97	0930			
		4-9-97	0915			
59	HEL-10	4-8-97	0930			
		4-9-97	0930			
60	FP-10	4-8-97	1010			
		4-9-97	1005			
61	BOR-10	4-8-97	1035			
		4-9-97	1035			
62	ARB-10	4-8-97	1230			
		4-9-97	1225			
63	SS-11	4-9-97	0915		Windy	
		4-10-97	0920			
64	SS-11D	4-9-97	0915			
		4-10-97	0920			
65	HEL-11	4-9-97	0930			
		4-10-97	0945			
66	HEL-11D	4-9-97	0930			
		4-10-97	0945			

**Project: Aldicarb Ambient**  
**Project #: C97-004**

166



**LOG BOOK**  
 Project: Aldicarb Ambient - KERN  
 Project #: C97-004 - 6

Log Number	Sample ID	Date	Time	Comments	weather o = overcast pc = partly cloudy k = clear   taken by	
79	WAS1	6/10/97	0925	WASCO H.S.	K	KEM
		6/11/97	0935			
80	M/F1	6/10/97	1011	McFarland		
		6/11/97	0900			
81	MAP1	6/10/97	1054	Maple School		
		6/11/97	1000			
82	SEV1	6/10/97	1026	S. Little Lake School + Poplar		
		6/11/97	1032			
83	BAK1	6/10/97	1105	ARB - Bakersfield	x	v
		6/11/97	1105			
84	NAS2	6/12/97	0935	RoTo 10	K	LDT
		6/12/97	0940			
85	WAS2D	6/11/97	0935	RoTo 20		
		6/12/97	0940			
86	MCF2	6/11/97	0900	RoTo 3		
		6/12/97	0915			
87	MCF2D	6/11/97	0920	RoTo 23		
		6/12/97	0915			
88	MAP2	6/11/97	1000	RoTo 2		
		6/12/97	1010			
89	MAP2D	6/11/97	1000	RoTo 25		
		6/12/97	1112			
90	SEV2	6/11/97	1022	RoTo 26		
		6/12/97	1030			
91	SEV2D	6/11/97	1022	RoTo 30		
		6/12/97	1030			
92	BAK2	6/11/97	1105	RoTo 21		
		6/12/97	1115			
93	BAK2D	6/11/97	1105	RoTo 24		
		6/12/97	1115			
94	BLNA	6/12/97	0940	WASCO H.S.		
95	WAS3	6/12/97	0940			
		6/13/97	1045			
96	MCF3	6/12/97	0915			
		6/13/97	1115			
97	MAP3	6/12/97	1019			
		6/13/97	1035			
98	SEV3	6/12/97	1030		O	
		6/13/97	1015			
99	BAK3	6/12/97	1115		O	v
		6/13/97	0935			

**LOG BOOK**  
Project: Aldicarb Ambient  
Project #: C97-004

Log Number	Sample ID	Date	Time	Comments	weather o = overcast pc = partly cloudy k = clear   taken by	
100	MCF4	6/16/97	1220	Rot. # 3	K/K	RJ
		6/17/97	950			
101	WAS4	6/16/97	1235	Rot. # 10	K/K	RJ
	MAP4	6/17/97	1020			
102	MAP4	6/16/97	1155	Rot. # 25	K/K	RJ
		6/17/97	1043			
103	SEV4	6/16/97	1310	Rot. # 30	K/K	RJ
		6/17/97	1100			
104	BAK4	6/16/97	1357	Rot. # 21	K/K	RJ
		6/17/97	1215			
105	MCF5	6/17/97	0950	Rot. # 3	K/PC	RJ
		6/18/97	1020			
106	WAS5	6/17/97	1020	Rot. # 10	K/PC	
		6/18/97	1050			
107	MAP5	6/17/97	1043	Rot. # 25	K/PC	
		6/18/97	1115			
108	SEV5	6/17/97	1100	Rot. # 30	K/PC	
		6/18/97	1130			
109	BAK5	6/17/97	1215	Rot. # 21	K/PC	
		6/18/97	1238			
110	MCF6	6/19/97	1020	Rot. # 3		
		6/20/97	1230			
111	MCF6-D	6/19/97	1020			
		6/20/97	1230			
112	WAS6	6/19/97	1050	Rot. # 10	PC/K	
		6/20/97	1037			
113	WAS6-D	6/19/97	1050	Rot. # 20	PC/K	
		6/20/97	1037			
114	MAP6	6/19/97	1115	Rot. # 25	PC/K	
		6/20/97	1057			
115	MAP6-D	6/19/97	1115	Rot. # 2	PC/K	
		6/20/97	1057			
116	SEV6	6/19/97	1130	Rot. # 30	PC/K	
		6/20/97	1112			
117	SEV6-D	6/19/97	1130	Rot. # 26	PC/K	
		6/20/97	1112			
118	BAK6	6/19/97	1238	Rot. # 21	PC/K	
		6/20/97	1147			
119	BAK6-D	6/19/97	1238	Rot. # 24	PC/K	
		6/20/97	1147			
120	MCF7	6/20/97	1230	Rot. # 3	K/K	
		6/21/97	0948			
121	WAS7	6/20/97	1037	Rot. # 10	K/K	
		6/21/97	0915			

**LOG BOOK**  
Project: Aldicarb Ambient  
Project #: C97-004

Log Number	Sample ID	Date	Time	Comments	weather o = overcast pc = partly cloudy k = clear   taken by	
122	MAP-7	6/20/97	1057	Rot #25	K/K	RJ
		6/21/97	0857			
123	SEV-7	6/20/97	1112	Rot #30	K/K	↓
		6/21/97	0840			
124	BAK-7	6/20/97	1147	Rot #24	K/K	↓
		6/21/97	0745			
125	Blank 1	6/19/97	1130			RJ
126	MCF-8	6-23-97	1100		K	LOT
		6-24-97	0910			
127	WAS-8	6-23-97	1130		(	(
		6-24-97	0935			
128	MAP-8	6-23-97	1150			
		6-24-97	0955			
129	SEI-8	6-23-97	1205			
		6-24-97	0915			
130	BAK-8	6-23-97	1300			
		6-24-97	1000			
131	BAK-8S1	6-23-97	1300	Rot 24 Spike		
		6-24-97	1000			
132	BAK-8S2	6-23-97	1300	Rot 29 Spike		
		6-24-97	1000			
133	BAK-8S1	6-23-97	1300	Trip Spike Baked		
134	BAK-8S2	6-23-97	1300	"		
135	BAK-8S3	6-23-97	1300	"		
136	BAK-8S4	6-23-97	1300	"		
137	BAK-8S5	6-23-97	1300	"		
138	MCF-9	6-24-97	0900			
		6-25-97	0900			
139	WAS-9	6-24-97	0935			
		6-25-97	0935			
140	MAP-9	6-24-97	0955			
		6-25-97	0955			
141	SEI-9	6-24-97	0915			
		6-25-97	1015			
142	BAK-9	6-24-97	1000			
		6-25-97	1100			
143	BAK-9S3	6-24-97	1000	Spike Rot 24	✓	✓
		6-25-97	1130			

**LOG BOOK**  
Project: Aldicarb Ambient  
Project #: C97-004

Log Number	Sample ID	Date	Time	Comments	weather o = overcast pc = partly cloudy k = clear   taken by	
144	BAK-954	6-24-97	1000	Spikes ROTO 29	K	LOT
		6-25-97	1100			
145	MCF-10	6-25-97	0900			
		6-26-97	0900			
146	MCF-100	6-25-97	0900			
		6-26-97	0900			
147	WAS-10	6-25-97	0930			
		6-26-97	0940			
148	WAS-100	6-25-97	0930			
		6-26-97	0940			
149	MAP-10	6-25-97	0955			
		6-26-97	1000			
150	MAP-100	6-25-97	0955			
		6-26-97	1000			
151	SEV-10	6-25-97	1015			
		6-26-97	1025			
152	SEV-100	6-25-97	1015			
		6-26-97	1025			
153	BAK-10	6-25-97	1100			
		6-26-97	1100			
154	BAK-100	6-25-97	1100			
		6-26-97	1100			
155	BLFA-	6-25-97	0900	Mc Fa Land		
156	BAK-1055	6-25-97	1100			
		6-26-97	1100			
157	MCF-11	6-26-97	0900			
		6-27-97	1135			
158	WAS-11	6-26-97	0940			
		6-27-97	1110			
159	MAP-11	6-26-97	0955			
		6-27-97	1040			
160	SEV-11	6-26-97	1025			
		6-27-97	1015			
161	BAK-11	6-26-97	1100			
		6-27-97	0930			

APPENDIX VI

ALDICARB APPLICATION # 1  
METEOROLOGICAL DATA

# ALDICARB APPLICATION #1 METEOROLOGICAL DATA (15min. averages)

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	83	1330	0.1	77.9	1007	48	282
1997	83	1345	0.5	78.6	1007	47	293
1997	83	1400	0.0	78.7	1007	43	273
1997	83	1415	0.0	80.9	1007	43	241
1997	83	1430	0.0	80.9	1007	43	202
1997	83	1445	0.0	79.5	1006	48	319
1997	83	1500	0.0	78.9	1006	49	309
1997	83	1515	0.0	79.2	1006	51	314
1997	83	1530	0.0	78.7	1006	49	297
1997	83	1545	0.0	79.3	1006	48	290
1997	83	1600	0.0	79.8	1006	48	309
1997	83	1615	0.0	79.3	1006	47	310
1997	83	1630	0.0	79.0	1005	48	314
1997	83	1645	0.0	78.6	1005	47	304
1997	83	1700	0.0	79.2	1005	47	279
1997	83	1715	0.0	79.2	1005	51	323
1997	83	1730	0.0	78.4	1005	52	313
1997	83	1745	0.0	77.4	1006	56	308
1997	83	1800	0.0	75.4	1006	61	288
1997	83	1815	0.0	73.6	1006	64	297
1997	83	1830	0.0	72.0	1006	67	302
1997	83	1845	0.0	70.6	1006	70	297
1997	83	1900	0.0	69.9	1006	71	291
1997	83	1915	0.0	69.3	1006	72	319
1997	83	1930	0.0	69.3	1006	70	319
1997	83	1945	0.0	68.7	1006	71	325
1997	83	2000	0.0	68.4	1007	71	304
1997	83	2015	0.0	68.1	1007	71	295
1997	83	2030	0.0	66.9	1007	74	301
1997	83	2045	0.0	66.4	1007	76	296
1997	83	2100	0.0	65.8	1007	76	290
1997	83	2115	0.0	64.3	1007	78	257
1997	83	2130	0.0	63.2	1007	80	271
1997	83	2145	0.0	62.7	1007	79	273
1997	83	2200	0.0	62.3	1007	76	265
1997	83	2215	0.0	61.8	1007	75	273
1997	83	2230	0.0	62.0	1007	74	272
1997	83	2245	0.0	61.9	1007	75	283
1997	83	2300	0.0	61.8	1007	73	306
1997	83	2315	0.0	61.6	1007	73	339
1997	83	2330	0.0	61.4	1007	75	220
1997	83	2345	0.0	61.5	1007	74	149

**ALDICARB APPLICATION #1 METEOROLOGICAL DATA (15min. averages)**

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	84	0	0.0	61.3	1008	77	95
1997	84	15	0.0	59.3	1008	87	150
1997	84	30	0.0	58.9	1008	88	133
1997	84	45	0.0	58.6	1008	85	213
1997	84	100	0.0	58.9	1008	82	221
1997	84	115	0.0	58.8	1008	82	183
1997	84	130	0.0	58.2	1008	84	156
1997	84	145	0.0	58.0	1008	88	115
1997	84	200	0.0	57.7	1008	86	151
1997	84	215	0.0	58.6	1008	83	211
1997	84	230	0.0	57.8	1008	86	209
1997	84	245	0.0	57.1	1008	88	124
1997	84	300	0.0	56.6	1008	94	121
1997	84	315	0.0	56.2	1008	93	129
1997	84	330	0.0	56.3	1008	93	159
1997	84	345	0.0	56.4	1008	92	159
1997	84	400	0.0	56.2	1008	92	189
1997	84	415	0.0	56.1	1008	92	171
1997	84	430	0.0	56.0	1008	94	166
1997	84	445	0.0	55.6	1008	94	165
1997	84	500	0.0	55.2	1008	95	138
1997	84	515	0.0	54.8	1008	96	80
1997	84	530	0.0	54.3	1008	97	79
1997	84	545	0.0	54.3	1008	98	95
1997	84	600	0.0	54.8	1009	96	96
1997	84	615	0.0	54.9	1009	96	108
1997	84	630	0.0	54.9	1009	96	109
1997	84	645	0.0	56.0	1009	95	120
1997	84	700	0.0	57.7	1009	93	125
1997	84	715	0.0	59.0	1009	91	110
1997	84	730	0.0	60.8	1010	88	137
1997	84	745	0.0	62.8	1010	85	156
1997	84	800	0.0	63.9	1010	82	144
1997	84	815	0.0	65.1	1010	81	149
1997	84	830	0.0	66.7	1010	79	114
1997	84	845	0.0	67.2	1010	79	103
1997	84	900	0.0	68.0	1010	77	105
1997	84	915	0.1	69.1	1010	75	112
1997	84	930	0.0	70.5	1010	74	101
1997	84	945	0.0	72.2	1010	72	112
1997	84	1000	0.0	73.0	1010	69	89
1997	84	1015	0.0	75.2	1010	67	109

# ALDICARB APPLICATION #1 METEOROLOGICAL DATA (15min. averages)

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	84	1030	0.0	75.9	1010	66	107
1997	84	1045	0.0	78.4	1010	65	86
1997	84	1100	0.0	78.1	1010	61	123
1997	84	1115	0.0	79.5	1010	59	139
1997	84	1130	0.0	80.6	1010	55	124
1997	84	1145	0.0	79.4	1010	57	89
1997	84	1200	0.0	79.2	1009	57	105
1997	84	1215	0.0	80.2	1009	56	137
1997	84	1230	0.0	81.1	1009	54	125
1997	84	1245	0.0	81.0	1009	54	92
1997	84	1300	0.0	84.0	1009	52	152
1997	84	1315	0.0	83.7	1008	50	195
1997	84	1330	0.0	83.6	1008	50	135
1997	84	1345	0.0	82.2	1008	48	146
1997	84	1400	0.0	83.6	1007	47	161
1997	84	1415	0.0	82.9	1007	47	161
1997	84	1430	0.0	83.7	1007	46	165
1997	84	1445	0.0	84.4	1007	46	124
1997	84	1500	0.0	83.8	1006	48	110
1997	84	1515	0.0	84.0	1006	47	103
1997	84	1530	0.0	85.0	1006	46	119
1997	84	1545	0.0	84.7	1006	45	114
1997	84	1600	0.0	85.0	1006	46	62
1997	84	1615	0.0	85.5	1006	46	99
1997	84	1630	0.0	86.0	1006	45	97
1997	84	1645	0.0	85.1	1006	48	49
1997	84	1700	0.0	84.5	1006	50	25
1997	84	1715	0.0	83.4	1006	53	20
1997	84	1730	0.0	82.4	1006	57	24
1997	84	1745	0.0	81.2	1006	61	19
1997	84	1800	0.0	79.7	1006	64	25
1997	84	1815	0.0	77.8	1006	65	29
1997	84	1830	0.0	75.8	1006	68	18
1997	84	1845	0.0	74.7	1006	69	28
1997	84	1900	0.0	74.1	1006	69	32
1997	84	1915	0.0	73.5	1006	70	29
1997	84	1930	0.0	72.9	1007	72	34
1997	84	1945	0.0	72.9	1007	72	33
1997	84	2000	0.0	71.7	1007	77	201
1997	84	2015	0.0	71.0	1007	72	341
1997	84	2030	0.0	71.3	1007	68	323
1997	84	2045	0.0	69.5	1007	78	253



**ALDICARB APPLICATION #1 METEOROLOGICAL DATA (15min. averages)**

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	84	2100	0.0	68.4	1008	77	253
1997	84	2115	0.0	67.7	1008	77	266
1997	84	2130	0.0	67.6	1008	77	280
1997	84	2145	0.0	67.6	1008	75	289
1997	84	2200	0.0	67.4	1008	76	280
1997	84	2215	0.0	66.8	1008	77	301
1997	84	2230	0.0	66.8	1007	76	311
1997	84	2245	0.0	66.2	1007	79	311
1997	84	2300	0.0	65.6	1007	80	306
1997	84	2315	0.0	65.9	1007	78	310
1997	84	2330	0.0	65.4	1007	79	303
1997	84	2345	0.0	64.7	1007	80	301
1997	85	0	0.0	63.8	1007	82	306
1997	85	15	0.0	63.1	1007	83	309
1997	85	30	0.0	62.2	1007	86	317
1997	85	45	0.0	61.8	1007	85	346
1997	85	100	0.0	61.8	1007	84	337
1997	85	115	0.0	61.7	1007	84	305
1997	85	130	0.0	61.5	1007	84	217
1997	85	145	0.0	61.2	1007	84	333
1997	85	200	0.0	61.0	1007	84	329
1997	85	215	0.0	60.3	1007	86	325
1997	85	230	0.0	59.5	1007	88	273
1997	85	245	0.0	58.4	1007	90	325
1997	85	300	0.0	58.6	1007	89	144
1997	85	315	0.0	58.6	1007	89	26
1997	85	330	0.0	58.4	1007	92	19
1997	85	345	0.0	57.7	1007	91	17
1997	85	400	0.0	57.9	1007	89	123
1997	85	415	0.0	57.5	1007	88	261
1997	85	430	0.0	57.7	1007	89	228
1997	85	445	0.0	56.9	1007	93	230
1997	85	500	0.0	55.7	1007	95	237
1997	85	515	0.0	55.4	1007	95	237
1997	85	530	0.0	55.3	1007	96	227
1997	85	545	0.0	54.8	1007	96	266
1997	85	600	0.0	54.6	1007	97	312
1997	85	615	0.0	55.5	1007	96	300
1997	85	630	0.0	56.0	1007	95	282
1997	85	645	0.0	57.0	1007	94	183
1997	85	700	0.0	58.3	1008	92	282
1997	85	715	0.0	60.0	1008	89	297

**ALDICARB APPLICATION #1 METEOROLOGICAL DATA (15min. averages)**

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	85	730	0.0	61.7	1008	86	323
1997	85	745	0.0	62.8	1008	83	332
1997	85	800	0.0	63.8	1008	80	318
1997	85	815	0.0	64.5	1008	80	328
1997	85	830	0.1	65.7	1008	79	309
1997	85	845	0.0	68.0	1008	78	200
1997	85	900	0.0	69.2	1008	76	86
1997	85	915	0.0	71.7	1008	73	67
1997	85	930	0.0	73.0	1008	73	282
1997	85	945	0.0	75.5	1008	71	103
1997	85	1000	0.0	76.7	1008	69	262
1997	85	1015	0.0	76.8	1008	65	122
1997	85	1030	0.0	78.3	1008	65	237
1997	85	1045	0.0	76.8	1008	65	212
1997	85	1100	0.0	79.4	1008	63	119
1997	85	1115	0.0	81.2	1008	61	148
1997	85	1130	0.0	79.1	1007	61	56
1997	85	1145	0.0	80.0	1007	61	129
1997	85	1200	0.0	81.9	1007	58	60
1997	85	1215	0.0	82.5	1007	58	234
1997	85	1230	0.0	82.3	1007	57	254
1997	85	1245	0.0	84.7	1006	53	238
1997	85	1300	0.0	85.5	1006	43	124
1997	85	1315	0.0	84.6	1006	44	172
1997	85	1330	0.0	85.4	1005	45	164
1997	85	1345	0.0	86.0	1005	43	258
1997	85	1400	0.0	85.9	1005	43	206
1997	85	1415	0.0	84.8	1005	45	111
1997	85	1430	0.0	85.7	1004	45	200
1997	85	1445	0.0	86.6	1004	43	242
1997	85	1500	0.0	86.9	1004	42	68
1997	85	1515	0.0	88.5	1004	41	139
1997	85	1530	0.0	86.9	1004	41	115
1997	85	1545	0.0	87.2	1003	38	266
1997	85	1600	0.0	86.0	1003	39	114
1997	85	1615	0.0	85.9	1003	37	192
1997	85	1630	0.0	86.0	1003	38	53
1997	85	1645	0.0	86.2	1003	39	70
1997	85	1700	0.0	85.5	1003	39	31
1997	85	1715	0.0	85.3	1003	39	281
1997	85	1730	0.0	84.4	1003	40	325
1997	85	1745	0.0	82.1	1003	44	333

**ALDICARB APPLICATION #1 METEOROLOGICAL DATA (15min. averages)**

<b>Year</b>	<b>Julian Date</b>	<b>Time</b>	<b>Wind Speed (mph)</b>	<b>Temp. (F)</b>	<b>Barometric Pressure (hPa)</b>	<b>Relative Humidity (%)</b>	<b>Wind Direction (degrees relative to geo. N)</b>
1997	85	1800	0.0	79.9	1003	48	330
1997	85	1815	0.0	78.2	1003	50	335
1997	85	1830	0.0	76.5	1003	55	331
1997	85	1845	0.0	75.6	1003	55	331
1997	85	1900	2.3	75.3	1003	49	328
1997	85	1915	3.4	74.3	1004	44	325
1997	85	1930	3.5	73.5	1004	42	321
1997	85	1945	7.7	72.9	1004	42	317
1997	85	2000	9.9	71.9	1004	42	310
1997	85	2015	9.1	70.6	1004	45	312

APPENDIX VII

ALDICARB APPLICATION # 2  
METEOROLOGICAL DATA

# ALDICARB APPLICATION #2 METEOROLOGICAL DATA (15 min. averages)

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	174	1316	4.1	84.8	1005	34	285
1997	174	1331	1.7	85.4	1005	34	325
1997	174	1346	1.8	85.4	1005	33	293
1997	174	1401	1.4	86.0	1005	33	295
1997	174	1416	0.2	86.8	1004	31	327
1997	174	1431	1.0	86.4	1004	29	229
1997	174	1446	0.6	86.4	1004	27	285
1997	174	1501	0.0	87.1	1004	27	323
1997	174	1516	0.1	87.3	1004	27	313
1997	174	1531	0.5	87.2	1004	28	320
1997	174	1546	0.5	87.5	1004	27	318
1997	174	1601	0.1	87.3	1004	27	311
1997	174	1616	0.1	87.7	1004	25	301
1997	174	1631	0.1	87.9	1003	25	291
1997	174	1646	0.0	88.0	1003	25	255
1997	174	1701	0.0	88.0	1003	24	251
1997	174	1716	0.0	88.0	1003	24	297
1997	174	1731	0.0	87.5	1003	23	324
1997	174	1746	0.0	87.6	1003	24	273
1997	174	1801	0.0	87.4	1003	25	336
1997	174	1816	0.0	87.4	1003	27	312
1997	174	1831	0.0	87.0	1003	29	311
1997	174	1846	0.0	86.6	1003	30	315
1997	174	1901	0.0	85.7	1003	33	323
1997	174	1916	0.0	85.0	1003	35	322
1997	174	1931	0.0	83.5	1003	36	317
1997	174	1946	0.0	81.3	1003	41	307
1997	174	2001	0.0	79.0	1003	45	301
1997	174	2016	0.0	76.5	1003	47	284
1997	174	2031	0.0	74.1	1003	52	273
1997	174	2046	0.0	73.1	1003	53	277
1997	174	2101	0.0	71.0	1003	58	283
1997	174	2116	0.0	69.1	1003	62	277
1997	174	2131	0.0	68.4	1004	63	274
1997	174	2146	0.0	67.7	1004	65	281
1997	174	2201	0.0	66.6	1004	66	278
1997	174	2216	0.0	65.8	1004	67	274
1997	174	2231	0.0	64.6	1004	69	270
1997	174	2246	0.0	64.1	1004	68	271
1997	174	2301	0.0	64.0	1004	66	281
1997	174	2316	0.0	63.7	1004	64	284

# ALDICARB APPLICATION #2 METEOROLOGICAL DATA (15 min. averages)

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	174	2331	0.0	62.8	1004	66	284
1997	174	2346	0.0	61.6	1004	69	284
1997	175	1	0.0	60.8	1004	70	290
1997	175	16	0.0	60.1	1004	72	288
1997	175	31	0.0	59.0	1004	74	321
1997	175	46	0.0	59.2	1004	73	337
1997	175	101	0.0	58.5	1004	77	28
1997	175	116	0.0	57.3	1004	81	53
1997	175	131	0.0	56.9	1004	82	12
1997	175	146	0.0	57.7	1004	80	99
1997	175	201	0.0	57.9	1004	79	123
1997	175	216	0.0	58.2	1003	77	329
1997	175	231	0.0	58.3	1003	77	195
1997	175	246	0.0	58.1	1003	78	79
1997	175	301	0.0	57.3	1003	80	301
1997	175	316	0.0	58.4	1003	78	342
1997	175	331	0.0	58.0	1003	79	313
1997	175	346	0.0	57.5	1003	80	335
1997	175	401	0.0	57.4	1003	81	341
1997	175	416	0.0	57.4	1003	81	340
1997	175	431	0.0	57.6	1003	81	333
1997	175	446	0.0	57.6	1003	79	337
1997	175	501	0.0	58.2	1003	77	296
1997	175	516	0.0	55.9	1003	82	99
1997	175	531	0.0	54.6	1003	85	213
1997	175	546	0.0	54.5	1003	85	181
1997	175	601	0.0	54.7	1004	86	211
1997	175	616	0.0	54.2	1004	89	192
1997	175	631	0.0	56.7	1004	87	213
1997	175	646	0.0	59.1	1004	86	197
1997	175	701	0.0	60.2	1004	87	200
1997	175	716	0.0	61.2	1004	83	216
1997	175	731	0.0	63.4	1004	77	251
1997	175	746	0.0	66.1	1004	71	240
1997	175	801	0.0	67.6	1004	66	255
1997	175	816	0.0	69.8	1004	64	273
1997	175	831	0.0	72.8	1004	58	286
1997	175	846	0.0	74.8	1004	54	302
1997	175	901	0.1	76.3	1004	50	282
1997	175	916	0.5	76.1	1005	49	272
1997	175	931	0.0	77.4	1005	48	261

# ALDICARB APPLICATION #2 METEOROLOGICAL DATA (15 min. averages)

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	175	946	0.0	78.8	1004	45	278
1997	175	1001	0.0	79.9	1004	40	289
1997	175	1016	1.0	81.4	1004	36	300
1997	175	1031	2.2	81.7	1004	35	322
1997	175	1046	0.8	83.7	1004	35	309
1997	175	1101	2.1	83.4	1004	34	312
1997	175	1116	0.4	83.8	1004	33	307
1997	175	1131	0.0	84.8	1004	34	304
1997	175	1146	0.1	85.5	1004	32	318
1997	175	1201	0.0	86.4	1004	32	285
1997	175	1216	0.0	86.7	1004	34	271
1997	175	1231	0.0	87.2	1004	33	250
1997	175	1246	0.0	87.3	1004	32	313
1997	175	1301	0.0	87.9	1003	32	269
1997	175	1316	0.0	88.2	1003	30	255
1997	175	1331	0.0	88.7	1003	32	257
1997	175	1346	0.0	90.3	1003	28	283
1997	175	1401	0.0	90.4	1003	29	186
1997	175	1416	0.0	91.0	1003	30	228
1997	175	1431	0.0	91.5	1003	31	228
1997	175	1446	0.0	91.4	1003	28	203
1997	175	1501	0.0	91.4	1002	30	270
1997	175	1516	0.0	92.6	1002	24	314
1997	175	1531	0.0	93.7	1002	24	298
1997	175	1546	0.0	93.0	1002	23	295
1997	175	1601	0.0	93.3	1002	22	285
1997	175	1616	0.0	93.7	1001	24	243
1997	175	1631	0.0	93.4	1001	26	182
1997	175	1646	0.0	93.0	1001	24	268
1997	175	1701	0.0	93.1	1001	25	274
1997	175	1716	0.0	93.2	1001	25	302
1997	175	1731	0.0	92.8	1001	27	258
1997	175	1746	0.0	92.8	1001	26	322
1997	175	1801	0.0	92.7	1000	28	280
1997	175	1816	0.0	92.2	1000	28	297
1997	175	1831	0.0	92.4	1000	30	305
1997	175	1846	0.0	91.5	1000	32	341
1997	175	1901	0.0	90.6	1000	38	336
1997	175	1916	0.0	89.6	1000	45	328
1997	175	1931	0.0	88.0	1000	50	307
1997	175	1946	0.0	86.8	1000	54	325

**ALDICARB APPLICATION #2 METEOROLOGICAL DATA (15 min. averages)**

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	175	2001	0.0	85.0	1000	59	321
1997	175	2016	0.0	82.1	1000	62	298
1997	175	2031	0.0	79.2	1000	65	291
1997	175	2046	0.0	77.6	1000	66	286
1997	175	2101	0.0	76.2	1001	66	295
1997	175	2116	0.0	74.5	1001	68	298
1997	175	2131	0.0	73.6	1001	66	305
1997	175	2146	0.0	72.0	1001	70	291
1997	175	2201	0.0	70.5	1001	73	274
1997	175	2216	0.0	69.6	1001	74	279
1997	175	2231	0.0	69.1	1001	73	275
1997	175	2246	0.0	68.7	1001	72	268
1997	175	2301	0.0	67.9	1001	73	282
1997	175	2316	0.0	67.3	1001	72	306
1997	175	2331	0.0	67.2	1001	72	326
1997	175	2346	0.0	66.1	1001	75	347
1997	176	1	0.0	66.5	1001	73	335
1997	176	16	0.0	66.6	1001	74	319
1997	176	31	0.0	65.9	1001	74	308
1997	176	46	0.0	65.5	1001	74	308
1997	176	101	0.0	65.0	1001	74	324
1997	176	116	0.0	64.7	1001	73	340
1997	176	131	0.0	63.3	1001	77	88
1997	176	146	0.0	62.8	1001	77	237
1997	176	201	0.0	63.9	1001	72	303
1997	176	216	0.0	63.6	1001	73	304
1997	176	231	0.0	63.5	1000	72	341
1997	176	246	0.0	62.9	1000	73	342
1997	176	301	0.0	63.0	1000	72	346
1997	176	316	0.0	61.2	1000	77	166
1997	176	331	0.0	61.1	1000	75	322
1997	176	346	0.0	63.4	1000	70	315
1997	176	401	0.0	62.8	1000	72	311
1997	176	416	0.0	60.7	1000	78	276
1997	176	431	0.0	58.7	1000	82	119
1997	176	446	0.0	57.5	1000	85	236
1997	176	501	0.2	56.6	1000	86	129
1997	176	516	0.0	57.4	1001	83	242
1997	176	531	0.0	57.0	1001	84	181
1997	176	546	0.0	56.0	1001	87	206
1997	176	601	0.0	56.3	1001	85	231



# ALDICARB APPLICATION #2 METEOROLOGICAL DATA (15 min. averages)

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	176	616	0.0	57.5	1001	82	240
1997	176	631	0.0	59.1	1001	83	147
1997	176	646	0.0	59.7	1001	87	126
1997	176	701	0.0	62.5	1001	85	185
1997	176	716	0.0	64.1	1001	81	255
1997	176	731	0.0	65.2	1001	77	262
1997	176	746	0.0	67.5	1001	73	265
1997	176	801	0.0	70.4	1002	66	270
1997	176	816	0.1	72.2	1002	59	306
1997	176	831	0.0	74.6	1002	58	301
1997	176	846	0.1	76.3	1002	55	305
1997	176	901	0.0	78.1	1002	50	311
1997	176	916	0.0	80.3	1002	47	323
1997	176	931	0.0	81.2	1002	44	308
1997	176	946	0.0	82.9	1002	43	301
1997	176	1001	0.0	84.6	1002	41	315
1997	176	1016	0.0	85.7	1002	38	311
1997	176	1031	0.0	87.1	1002	32	322
1997	176	1046	0.0	89.2	1002	29	299
1997	176	1101	0.0	89.8	1002	28	317
1997	176	1116	0.0	89.6	1002	28	309
1997	176	1131	0.0	90.0	1002	29	312
1997	176	1146	0.0	90.9	1002	29	322
1997	176	1201	0.0	91.5	1002	28	304
1997	176	1216	0.0	93.0	1001	29	332
1997	176	1231	0.0	93.5	1001	28	324
1997	176	1246	0.0	93.2	1001	28	288
1997	176	1301	0.0	94.2	1001	28	309
1997	176	1316	0.0	94.7	1001	28	328
1997	176	1331	0.0	94.9	1001	27	278
1997	176	1346	0.0	95.6	1000	26	221
1997	176	1401	0.0	96.1	1000	26	292
1997	176	1416	0.0	96.2	1000	26	287
1997	176	1431	0.0	96.8	1000	28	319
1997	176	1446	0.0	95.8	1000	28	255
1997	176	1501	0.0	95.2	1000	27	232
1997	176	1516	0.0	95.4	1000	26	319
1997	176	1531	0.0	95.1	999	26	228
1997	176	1546	0.0	95.6	999	26	167
1997	176	1601	0.0	96.1	999	26	253
1997	176	1616	0.0	95.5	999	28	211

# ALDICARB APPLICATION #2 METEOROLOGICAL DATA (15 min. averages)

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	176	1631	0.0	95.8	999	28	232
1997	176	1646	0.0	95.8	999	28	237
1997	176	1701	0.0	95.7	999	28	190
1997	176	1716	0.0	96.2	998	27	345
1997	176	1731	0.0	96.9	998	27	308
1997	176	1746	0.0	96.5	998	27	336
1997	176	1801	0.0	95.7	998	29	299
1997	176	1816	0.0	95.0	998	30	274
1997	176	1831	0.0	95.3	998	34	301
1997	176	1846	0.0	94.9	998	35	322
1997	176	1901	0.0	94.3	998	39	322
1997	176	1916	0.0	92.5	998	39	316
1997	176	1931	0.0	90.6	998	46	303
1997	176	1946	0.0	88.9	998	46	301
1997	176	2001	0.0	86.8	998	51	306
1997	176	2016	0.0	83.6	998	57	276
1997	176	2031	0.0	81.0	998	61	278
1997	176	2046	0.0	79.7	998	61	290
1997	176	2101	0.0	77.9	998	63	299
1997	176	2116	0.0	76.7	999	65	302
1997	176	2131	0.0	74.7	999	72	281
1997	176	2146	0.0	74.0	999	70	293
1997	176	2201	0.0	73.7	999	69	308
1997	176	2216	0.0	72.6	999	70	303
1997	176	2231	0.0	71.4	999	73	291
1997	176	2246	0.0	71.0	999	72	295
1997	176	2301	0.0	71.0	999	70	305
1997	176	2316	0.0	71.8	999	67	293
1997	176	2331	0.0	71.0	999	67	312
1997	176	2346	0.0	69.5	1000	71	335
1997	177	1	0.0	69.6	1000	69	320
1997	177	16	0.0	71.1	1000	64	315
1997	177	31	0.0	70.3	1000	64	341
1997	177	46	0.0	68.8	1000	68	317
1997	177	101	0.0	69.8	1000	63	326
1997	177	116	0.1	69.7	1000	61	319
1997	177	131	0.0	68.7	1000	64	309
1997	177	146	0.0	67.0	1000	68	312
1997	177	201	0.0	66.2	1000	70	313
1997	177	216	0.0	65.6	1000	71	316
1997	177	231	0.0	65.0	1000	72	310

**ALDICARB APPLICATION #2 METEOROLOGICAL DATA (15 min. averages)**

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	177	246	0.0	63.6	1000	76	297
1997	177	301	0.0	63.9	1000	74	285
1997	177	316	0.5	64.5	1000	70	293
1997	177	331	1.4	65.2	1000	67	309
1997	177	346	1.2	64.6	1000	70	314
1997	177	401	0.0	63.0	1000	74	321
1997	177	416	0.0	62.0	1000	76	309
1997	177	431	1.0	62.5	1000	74	317
1997	177	446	0.1	62.1	1000	75	322
1997	177	501	0.0	60.8	1000	77	334
1997	177	516	0.5	61.0	1000	76	321
1997	177	531	0.7	60.7	1000	77	314
1997	177	546	2.3	60.6	1000	76	312
1997	177	601	3.0	61.0	1001	74	311
1997	177	616	0.5	61.1	1001	75	309
1997	177	631	0.0	61.8	1001	77	304
1997	177	646	0.9	63.4	1001	74	306
1997	177	701	1.6	66.2	1001	67	338
1997	177	807	3.1	68.8	1001	64	320
1997	177	822	3.5	74.2	1002	58	313
1997	177	837	4.8	75.9	1002	53	323
1997	177	852	4.8	77.3	1002	48	318
1997	177	907	3.8	79.0	1002	45	312
1997	177	922	5.4	79.7	1002	40	331
1997	177	937	5.6	80.0	1002	39	299
1997	177	952	4.8	80.7	1002	40	301
1997	177	1007	2.4	82.2	1002	38	279
1997	177	1022	2.2	83.3	1002	34	277
1997	177	1037	0.0	85.6	1002	37	255
1997	177	1052	0.0	87.3	1002	35	249
1997	177	1107	0.7	86.8	1002	30	317
1997	177	1122	1.2	86.8	1002	28	298
1997	177	1137	0.0	88.8	1002	28	314
1997	177	1152	0.3	88.3	1002	26	274
1997	177	1207	0.0	89.1	1002	25	300
1997	177	1222	0.0	90.2	1002	23	212
1997	177	1237	0.0	90.8	1002	23	290
1997	177	1252	0.0	91.4	1002	24	269
1997	177	1307	0.0	91.5	1001	24	213
1997	177	1322	0.0	91.8	1001	24	212
1997	177	1337	0.0	93.0	1001	24	231

# ALDICARB APPLICATION #2 METEOROLOGICAL DATA (15 min. averages)

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	177	1352	0.0	93.5	1001	24	299
1997	177	1407	0.0	94.4	1001	24	289
1997	177	1422	0.0	95.5	1001	24	321
1997	177	1437	0.0	96.2	1000	23	320
1997	177	1452	0.0	95.7	1000	24	327
1997	177	1507	0.0	95.7	1000	26	143
1997	177	1522	0.0	95.3	1000	26	256
1997	177	1537	0.0	96.0	1000	25	297
1997	177	1552	0.0	95.1	1000	27	215
1997	177	1607	0.0	95.4	999	28	255
1997	177	1622	0.0	94.6	999	26	302
1997	177	1637	0.0	95.2	999	25	337
1997	177	1652	0.0	94.1	999	25	275
1997	177	1707	0.0	93.2	999	27	331
1997	177	1722	0.0	93.2	999	28	311
1997	177	1737	0.0	93.3	999	28	324
1997	177	1752	0.0	91.0	999	28	311
1997	177	1807	0.0	88.2	999	30	304
1997	177	1822	0.0	86.7	999	34	309
1997	177	1837	0.0	85.9	999	36	306
1997	177	1852	0.0	85.8	999	42	307
1997	177	1907	0.0	85.5	999	41	313
1997	177	1922	0.0	84.4	999	41	306
1997	177	1937	0.0	83.6	999	41	319
1997	177	1952	0.0	82.6	1000	43	316
1997	177	2007	0.0	82.2	1000	45	309
1997	177	2022	0.0	81.5	1000	45	317
1997	177	2037	0.2	80.5	1000	43	317
1997	177	2052	0.0	78.4	1000	46	314
1997	177	2107	0.2	77.7	1000	42	310
1997	177	2122	1.3	77.6	1000	34	310
1997	177	2137	1.5	76.9	1000	31	306
1997	177	2152	0.1	75.5	1001	34	315
1997	177	2207	0.1	73.8	1001	39	312
1997	177	2222	0.1	73.0	1001	41	315
1997	177	2237	0.2	72.2	1001	43	313
1997	177	2252	1.3	71.5	1001	44	317
1997	177	2307	4.4	71.6	1001	42	318
1997	177	2322	1.5	70.6	1001	42	291
1997	177	2337	0.1	67.9	1001	50	293
1997	177	2352	0.2	66.8	1001	54	309

# ALDICARB APPLICATION #2 METEOROLOGICAL DATA (15 min. averages)

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	178	7	0.1	66.1	1001	55	312
1997	178	22	0.2	66.1	1001	54	318
1997	178	37	0.2	65.6	1001	55	327
1997	178	52	0.8	64.9	1001	57	323
1997	178	107	3.8	64.4	1001	57	329
1997	178	122	2.2	64.5	1001	56	300
1997	178	137	1.5	64.0	1001	56	302
1997	178	152	1.7	63.0	1001	58	328
1997	178	207	1.5	63.0	1001	57	321
1997	178	222	2.6	62.5	1001	57	317
1997	178	903	3.6	72.7	1004	43	311
1997	178	918	4.2	72.9	1004	43	253
1997	178	933	2.4	74.2	1004	43	295
1997	178	948	2.5	75.0	1004	42	233
1997	178	1003	1.7	75.9	1004	41	234
1997	178	1018	0.3	77.7	1004	40	295
1997	178	1033	0.2	79.3	1004	37	255
1997	178	1048	0.1	81.2	1004	36	288
1997	178	1103	0.0	83.1	1004	36	236
1997	178	1118	0.0	83.2	1004	32	278
1997	178	1133	0.0	83.6	1004	30	275
1997	178	1148	0.0	84.3	1004	26	300
1997	178	1203	0.0	85.0	1004	25	291
1997	178	1218	0.0	87.0	1004	23	270
1997	178	1233	0.0	87.5	1004	25	248
1997	178	1248	0.0	86.9	1004	25	57
1997	178	1303	0.0	87.2	1004	23	140
1997	178	1318	0.0	86.9	1004	26	127
1997	178	1333	0.0	87.0	1003	24	62
1997	178	1348	0.0	88.5	1003	22	89
1997	178	1403	0.0	89.0	1003	20	173
1997	178	1418	0.0	89.3	1003	19	86
1997	178	1433	0.0	89.6	1003	20	194
1997	178	1448	0.0	89.9	1003	21	89
1997	178	1503	0.0	90.5	1003	21	129
1997	178	1518	0.0	89.8	1003	20	93
1997	178	1533	0.0	89.7	1003	21	261
1997	178	1548	0.0	90.2	1003	20	144
1997	178	1603	0.0	89.8	1002	23	105
1997	178	1618	0.0	90.2	1002	23	214
1997	178	1633	0.0	89.9	1002	24	233

# ALDICARB APPLICATION #2 METEOROLOGICAL DATA (15 min. averages)

Year	Julian Date	Time	Wind Speed (mph)	Temp. (F)	Barometric Pressure (hPa)	Relative Humidity (%)	Wind Direction (degrees relative to geo. N)
1997	178	1648	0.0	89.9	1002	24	255
1997	178	1703	0.0	90.2	1002	22	281
1997	178	1718	0.0	89.0	1002	22	189
1997	178	1733	0.0	88.9	1002	23	278
1997	178	1748	0.0	89.0	1002	24	299
1997	178	1803	0.0	89.4	1002	23	313
1997	178	1818	0.0	89.0	1002	23	329
1997	178	1833	0.0	87.6	1002	23	322
1997	178	1848	0.0	86.4	1002	23	315
1997	178	1903	0.0	85.5	1002	22	312
1997	178	1918	0.0	84.4	1002	22	320
1997	178	1933	0.0	83.1	1002	24	309
1997	178	1948	0.0	81.5	1002	26	310
1997	178	2003	0.0	79.9	1002	29	324
1997	178	2018	0.0	78.1	1002	33	318
1997	178	2033	0.0	76.4	1002	37	320
1997	178	2048	0.0	75.1	1002	41	332
1997	178	2103	0.4	74.1	1002	44	327
1997	178	2118	3.0	73.5	1002	46	322
1997	178	2133	4.5	73.0	1002	47	316
1997	178	2148	4.6	72.4	1003	49	317
1997	178	2203	5.3	71.6	1003	51	322
1997	178	2218	4.8	70.8	1003	52	319
1997	178	2233	5.2	69.9	1003	54	319
1997	178	2248	5.7	69.0	1003	55	317
1997	178	2303	8.0	68.3	1003	56	309
1997	178	2318	6.9	67.6	1003	58	305
1997	178	2333	5.6	66.9	1003	58	315
1997	178	2348	6.1	66.1	1003	60	321
1997	179	3	4.1	65.3	1003	61	335
1997	179	18	0.2	63.9	1003	64	254
1997	179	33	1.4	62.2	1003	69	278
1997	179	48	3.6	62.2	1003	68	256
1997	179	103	2.6	61.6	1003	69	339
1997	179	118	1.7	60.7	1003	71	333
1997	179	133	1.8	60.1	1004	71	311
1997	179	148	1.0	59.5	1004	72	306
1997	179	859	3.9	63.1	1005	63	338
1997	179	914	6.2	67.5	1006	55	339